ESRF	**Experiment title:** Phonons in supercrystals of nanocrystals	**Experiment number**: HC-1702
Beamline: ID28	**Date of experiment**: from: Nov 27, 2014 to: Dec 03, 2014	**Date of report**: Feb 17, 2015 *Received at ESRF:*
Shifts: 18	**Local contact(s)**: Thomas FORREST	

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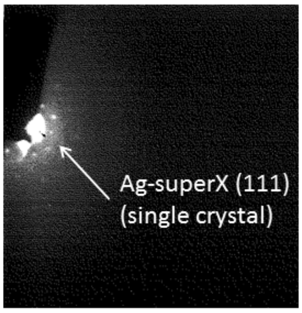
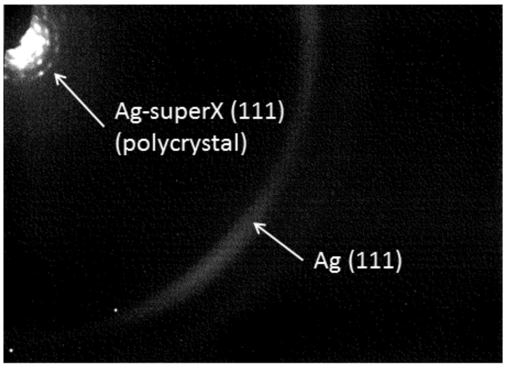
Report:

The experiment was performed in 2/3 filling mode at the Si(9 9 9), in order to favor the inelastic signal due to the smallness of the samples. Several samples of Ag supercrsytals (with nanocrystals diameters between 3.7 nm and 4.5 nm) were purposely grown for the IXS experiment. Most of the samples consisted of a collection of single facetted supercrystals of sizes between 1 and 10 µm, dispersed on a diamond substrate, while some samples consisted of supercrystalline films. All samples were characterized through a rotating anode GISAXS prior to the IXS experiment, to assess the supercrystallinity.

In order to run the inelastic scans, it was first advised to isolate a single supercrystal with a good diffraction pattern. Given the size of the supercrystals, such was not easy. Yet, using the PRL mount, we could identify for some samples regions on the diamond substrates that displayed big enough supercrystals, with well defined facets. The corresponding diffraction patterns, recorded with a CCD





camera placed ~20 cm downstream the sample turned satisfactory for some of them (see figure). From those successful samples, inelastic scans were performed. However, in the spite of the many attempts made, it was not possible to detect an inelastic signal. Two essential severe limitations were identified for our experiments:

(i) Due to the **large supercrystal lattice parameter** (a~10 nm), the dimensions of the **Brillouin zone** are **strongly reduced**. This requires to place the TTH arm extremely close to the direct beam (e.g. TTH=0.34° @ (½, ½,½)). So close to the direct beam, there remains significant residual scattering from either the supercrystal imperfections and/or from the direct beam leak-in (although care was taken to place beamstops at strategic locations). This situation turned out worse for supercrystalline films. Note that it is difficult to reach a crystalline quality for supercrystals which compares with that of atomic crystals so that it was impossible to work in higher order Brillouin zones.

(ii) The **typical size of the supercrystals (~5 µm)**, which is already quite significant for such objects, makes the amount of matter very weak for an IXS experiment (the x-ray attenuation length of Ag at 20 keV is 50 µm).

After 4 days of unsuccessfull attempts in obtaining an inelastic signal, it was decided to stop the experiment. Investigations to circumvent to the difficulties met are under way.
We thank the ID28 beamline staff for their valuable collaboration.