ESRF	**Experiment title:** In-situ Coherent X-ray Diffraction of Ensembles and Single Ir Nanoparticles during Catalytic Oxidation	**Experiment number**: HC-2608
Beamline: ID01	**Date of experiment**: from: 26.10.2016 to: 01.11.2016	**Date of report**:
Shifts: 18	**Local contact(s)**: Tobias U. Schülli and Marie-Ingrid Richard	*Received at ESRF:*

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Report:

We investigated substrate-supported Ir nanoparticle arrangements deposited at 800° C and 950° C and a single Pt nanoparticle, all grown by evaporation on (100)-oriented STO single crystal surfaces using coherent Bragg diffraction in a 8.5 keV energy X-ray beam focused to 150 nm × 300 nm. All samples were marked by e-beam induced deposition of Pt to re-localize the regions of interests containing suitable nanoparticles, as pre-selected by SEM.

For all samples we were able to localize the markers by using different contrasts, like absorption of the substrate Bragg peak or the Pt(111) crystal truncation rod signal, see Fig. 1.

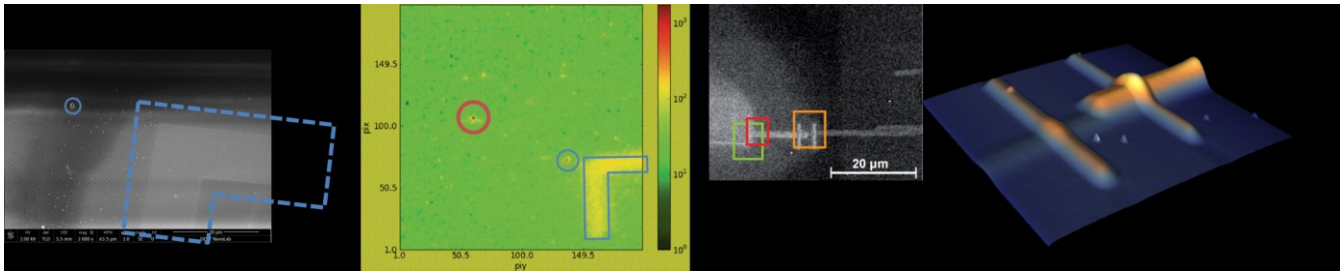

Fig. 1: Hierarchical markers written by e-beam induced deposition in the SEM to locate the pre-selected single Pt nanoparticle using the fast-scan K-MAP option at ID01. SEM (left) and Bragg Pt(111) crystal truncation rod image (center). Smaller markers were deposited after the beamtime to re-localize the Pt nanoparticle by AFM (right).

The intensity of the Ir Bragg peaks from the ~ 80 nm × 80 nm large and ~ 20 nm high Ir nanoparticles were too weak to analyze. With the new KB mirror setup at ID01 resulting in a higher intensity, it shall be feasible to obtain Ir Bragg scattering data in a future beamtime.

Furthermore, we were able to localize the isolated Pt nanoparticle (encircled in the center image of Fig. 1), and to collect a set of coherent Pt (111) Bragg diffraction data under continuous flow of Ar at RT for different sample tilts (eta) to map the full reciprocal space. The same data was collected after heating to approx. 100° C, repeating the same sample tilt scan. Subsequently, we changed the gas composition to pure CO and a 50:50 mixture of CO and O_2 while tracking the particle position and collecting each time a full data set in a sample tilt scan. For a given eta = 16.78, Fig. 2 displays raw 2D detector images of the Pt (111) Bragg peak for the catalytic steps during the experiment. The images clearly show the interference fringes arising from coherent single particle diffraction and indicate that during the course of the catalytic reaction subtle particle shape changes are taking place.

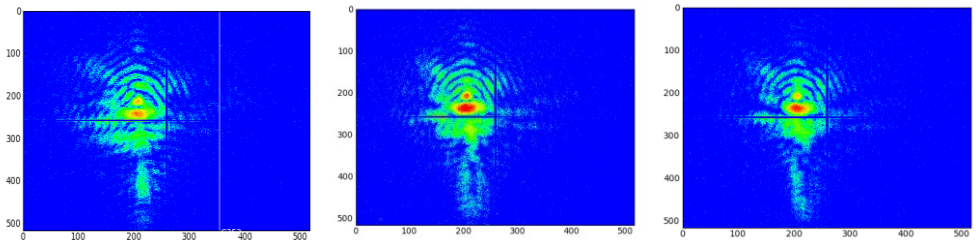

Fig. 2: 2D detector images of the coherently scattered Pt(111) Bragg peak from a single Pt nanoparticle in Ar (left), in CO atmosphere (center), and in a 50:50 mixture of CO and O_2 (right).

We are currently assembling the 3D reciprocal space information from the X-ray data for each of the 3 steps of the catalytic reaction (Fig. 3, left), and were able to re-locate and identify the isolated Pt nanoparticle that was analyzed at ID01 with the UHV-AFM, obtaining its surface topography (Fig. 3, right). Further data reconstruction is ongoing.

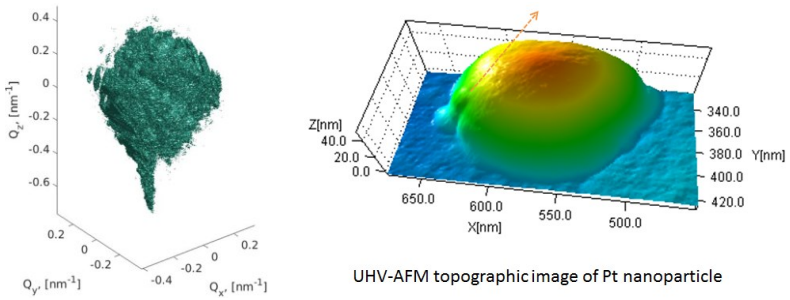

Fig. 3: 3D (left) reciprocal space iso-intensity lines obtained from an eta scan, and (right) UHV-AFM surface topography from exactly the same Pt nanoparticle after the re-localization at DESY NanoLab.

From the experiment HC-2608 at ID01 and the preliminary data analysis we conclude:

- We obtained one-to-one structure information from exactly the same single Pt nanoparticle at the beamtime ID01 and in the UHV-AFM at DESY NanoLab.
- We were able to track subtle particle shape changes of the Pt nanoparticle during catalytic reactions under mild conditions.
- The preliminary data analysis suggests shrinkage of the Pt nanoparticle size during the catalytic reaction.
- We successfully implemented, tested and utilized the "Advanced Nano-Object Transfer and Positioning", facilitating the easy and time-optimized re-localization of pre-selected nano-objects, an EU initiative funded in the framework of the H2020 program "Nanoscience Foundries an Fine Analysis" (NFFA), grant nr. 654360.

A first report is available at **#weekendusers Catalysis in a nanoparticle** (28-10-2016), a publication including the ESRF beamline local contacts as co-authors is in preparation.