ESRF	**Experiment title:** Operando coherent X-ray diffraction imaging of PtRh model catalysts	**Experiment number**: HC-3201
Beamline: ID01	**Date of experiment**: from: 22.11.2017 to: 28.11.2017	**Date of report**:
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Report:

Single PtRh nanoparticles (PtRh NP) on (100)-oriented SrTiO3 single crystal surfaces and single PtRh NPs on (0001)-Al_2O_3 single crystal surfaces have been investigated by coherent Bragg diffraction in an X-ray beam of 8.5 keV energy focused to 300 nm × 350 nm. The NPs were pre-selected in the SEM and subsequently marked by e-beam induced deposition of Pt markers. These hierarchical markers along with the "Advanced Nano-Object Transfer and Positioning" protocol developed in the framework of the EU funded program Nanoscience Foundries and Fine Analysis (NFFA) implemented at beamline ID01 enabled us to re-localize the pre-selected single PtRh NPs. For all samples, we were able to localize the markers by using different contrasts, like absorption of the substrate Bragg peak or the specular Pt(111) Bragg signal, see Fig. 1, and based on the marker positions, re-locate and analyze the pre-selected NPs.

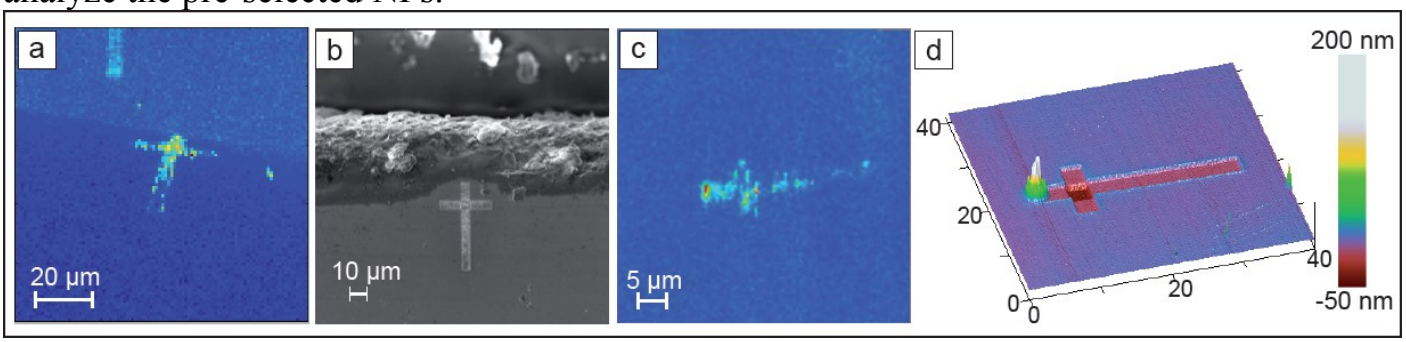

Fig. 1: Hierarchical markers to locate the pre-selected single PtRh nanoparticle. (a) K-MAP and (b) SEM image of the first marker created close to the edge of the sample. (c) K-MAP and (d) 3D AFM of markers of the 2nd hierarchy level.

We were able to re-localize the single PtRh NP for both samples and to collect a set of coherent Pt (111) Bragg diffraction data under continuous flow of Ar, Ar+CO and Ar+CO+O2 at 350º C (Fig. 2), while tracking the particle position and collecting each time a full data set in a sample tilt scan. Gas flow was 50 ml/min and pressure in the reactor was 0.1 bar during all the experiments. We collected the signal of the gases during the experiment by a mass spectrometer located in the exhaust gas line. Raw 2D detector images of the Pt(111) Bragg peak (eta = 17.98°) for the catalytic steps during the experiment are shown in Fig. 2. 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.

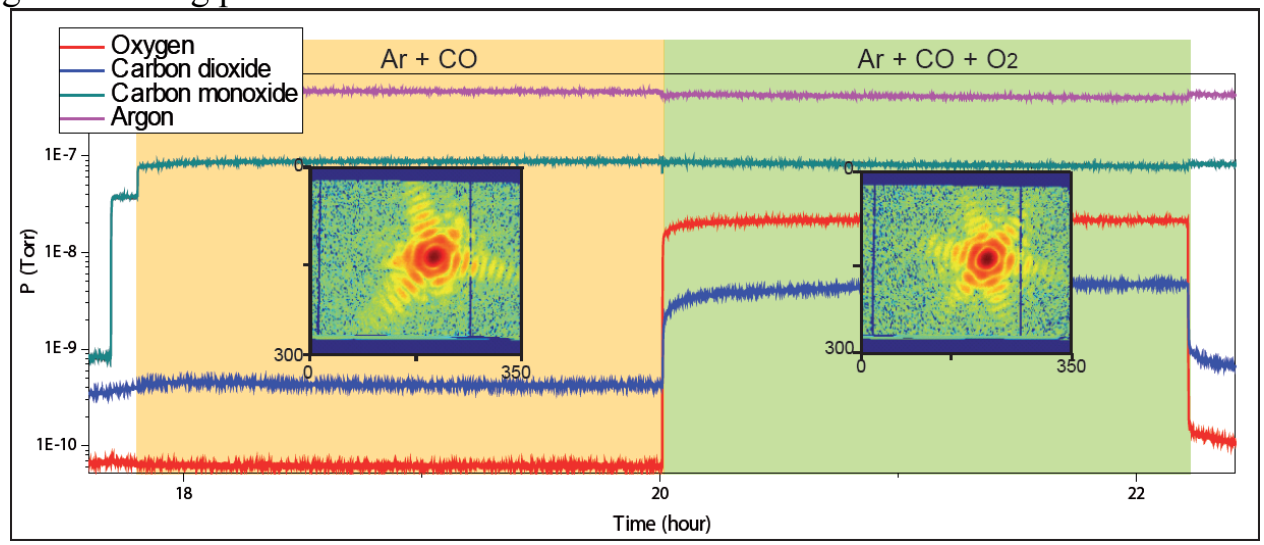

Fig. 2: 2D detector images of the coherently scattered Pt(111) Bragg peak from a single PtRh NP in Ar (white stripe on the left), in CO (+Ar) atmosphere (center, yellow), and in a 50:50 mixture of CO and O_2 (+Ar) atmosphere (right, green). The gas compositions in the exhaust gas line as detected by the mass spectrometer in the background.

We constructed the 3D reciprocal space information from the X-ray data for each of the steps of the catalytic reaction (Fig. 2). The quality of the data demonstrates that the data reconstruction will be feasible. We were able to re-locate and identify the single PtRh NP that was analyzed at ID01 by SEM and AFM, obtaining its surface topography to complete the study. Further data reconstruction and real space post-analysis at DESY NanoLab is ongoing.

From the experiment HC-3201 at ID01 and the preliminary data analysis we can conclude:

- We obtained one-to-one structure information from exactly the same single PtRh NP at ID01 beamline and DESY NanoLab.
- We have observed significant structural changes related to the catalytic process.
- We have performed the first catalyst imaging experiment on a single NP alloy under operation conditions.
- We are able to re-localize a pre-selected single PtRh NP at different Lab-based nanoscience instruments and at the ID01 nano-focus X-ray beamline thanks to our “Advanced Nano-Object Transfer and Positioning” protocol software, funded by the EU initiative in the framework of the H2020 program “Nanoscience Foundries and Fine Analysis” (NFFA), grant nr. 654360, and which is now implemented at ID01.
- A publication including the ESRF beamline local contacts as co-authors is in preparation.