ESRF	**Experiment title:** **In-situ Catalytic Structure-Activity Relationships of Single Pt Nanoparticles by Coherent X-Ray Diffraction Imaging.**	**Experiment number**: CH-4426
Beamline: ID01	**Date of experiment**: from: 8 February 2016 to: 16 February 2016	**Date of report**: 1 March 2016
Shifts: 18	**Local contact(s)**: Dr. Gilbert Chahine and Dr. Marie-Ingrid Richard	*Received at ESRF:*

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Report:

Our goal was to study the structural and catalytic properties of single Platinum (Pt) nanocrystals (NCs) using coherent X-ray diffraction imaging (CDI) in Bragg condition under *in situ* reaction conditions. The 3D intensity distribution in reciprocal space in the vicinity of a selected Bragg reflection contains information about the shape and strain states of the nanostructure; it provides crucial insight into the nature of nanoscale deformation following chemical stimuli, such as adsorption of gases.

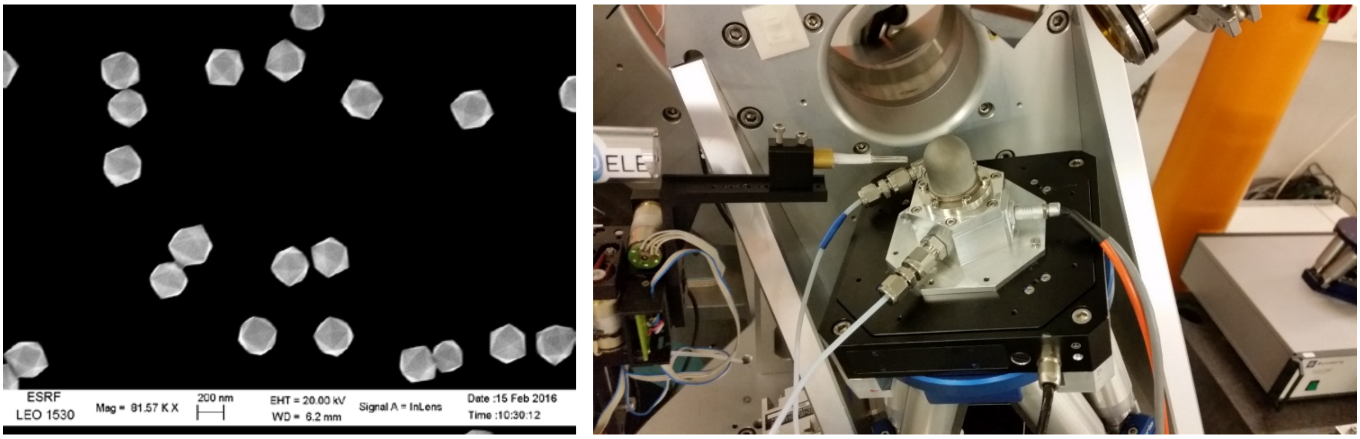

Fig. 1 *Left: Scanning electron microscopy (SEM) image of THH Pt nanocrystals. Right: Photograph of the new experimental furnace developed for gas-phase catalysis experiments.*

High-index tetrahexaedral (THH) Pt nanocrystals were were electrochemically synthesized at TU Eindhoven and at the electrochemical laboratory of ESRF (see Fig. 1). The THH NCs supported (fixed) on a glassy carbon substrate were investigated by coherent x-ray diffraction in Bragg condition using the nano-focused x-ray beam at the ID01 beamline. The diameter of the Pt NCs was varied from 50 to 400 nm. The required spatial resolution was obtained with a circular Fresnel Zone plate, giving a measured focal size of 170 × 450 nm^2 (vertical × horizontal) with a coherent illumination. A lightweight reactor compatible with CO, O_2 and H_2 gases (gas inlet/outlet and heater connection) has been developed for this experiment (see Fig. 1). Gas mixtures were controlled by a rack of mass flow controllers using He as carrier gas.

The nano-diffraction experiment was carried out at a beam energy of 8 keV, so that the **002** Pt Bragg peak

was accessible at a scattering angle of 23.269°. Isolated THH Pt particles have been located using the quicK-Mapping technique developed at beamline ID01 [1]. We succeeded to measure *single* Pt NCs using nano-focused coherent X-ray diffraction in a gas-phase environment. Reciprocal space maps (RSMs) were collected around the **002** Bragg reflection for different *single* THH Pt NCs using the two-dimensional (2D) Maxipix detector. We have carried out 3D coherent imaging of several *single* NCs as a function of the CO and O_2 partial pressures. We succeeded to follow the coherent diffraction pattern of single particles from room temperature until 200°C. As an example, Figure 2 displays the measured intensity at the maximum of the rocking-curve for a particle at a temperature of 200°C under He flow (left) and under CO oxidation conditions (right). CO and O_2 flows were 2.5 ml/min for each gas in 20 ml/min He carrier.

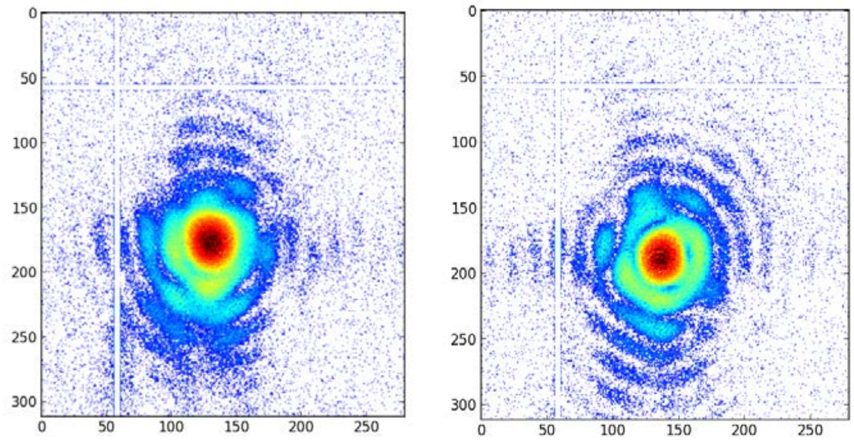

Fig. 2 *Detector image at the maximum of the rocking-curve for a THH Pt particle at a temperature of 200°C under He flow (left) and under CO oxidation conditions (2.5 ml/min CO + 2.5 ml/min* O_2*) (right).*

A change of the diffraction patterns as a function of gas mixture is observed in Fig. 2. The inversion of the coherent diffraction patterns is under way to recover the nanoparticle shape and strain at the different stages of the experiment. Figure 3 shows a detector image at the maximum of the rocking-curve for another THH Pt particle at a temperature of 200 °C. The q_y position of the diffraction peak (see Fig. 3 - right) also varies as a function of gas mixtures.

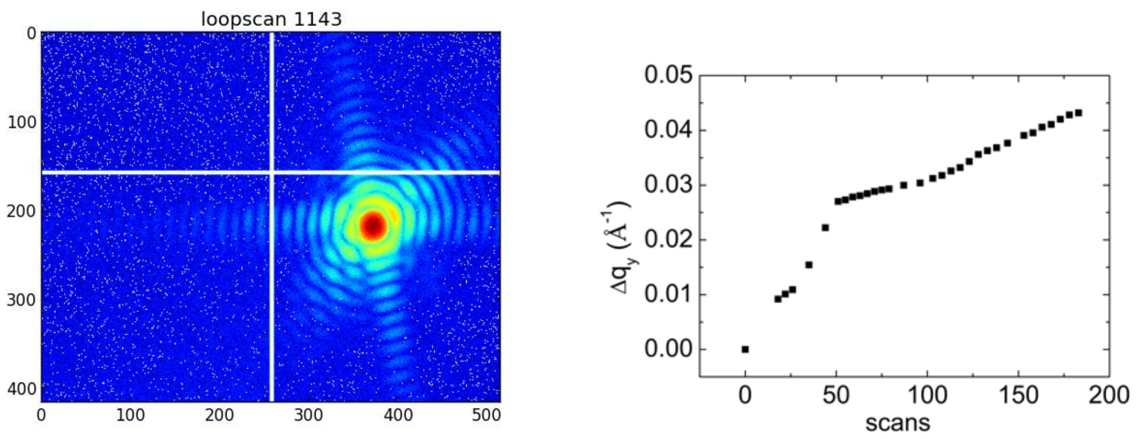

Fig. 3 *Detector image at the maximum of the rocking-curve for another THH Pt particle at a temperature of 200°C (left). Evolution of the* q_y *position of the diffraction peak as a function of scans (i.e. for different gas mixtures - right).*

References:
[1] G. A. Chahine, M.-I. Richard, R. A. Homs-Regojo, T. N. Tran-Caliste, D. Carbone, V. L. R. Jacques, R. Grifone, P. Boesecke, J. Katzer, I. Costina, H. Djazouli, T. Schroeder, and T. U. Schülli, J. Appl. Crystallogr. **47**, 762 (2014).