ESRF	**Experiment title:** In Situ Coherent X-Ray Diffraction Imaging of High-Index Faceted Platinum Particles	**Experiment number**: HC3270
Beamline: ID01	**Date of experiment**: from: November 15, 2017 to: November 21, 2017	**Date of report**: XXX
Shifts: 18	**Local contact(s)**: Dr. Marie-Ingrid Richard	*Received at ESRF:*

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Report:

Our goal was to study the structural and catalytic properties of individual (pure Pt and alloyed (Cu, Ni) Pt) nanoparticles (NPs) with controlled morphology at ID01 beamline using Bragg coherent x-ray diffraction imaging (Bragg CDI) during a catalytic reaction. We wanted to assess the influence of the morphological and structural (strain, defects, …) dynamical evolution of individual nanoparticles on their catalytic properties. A careful *in situ* analysis of the properties of the nanoparticles (size, shape, strain) is of essential importance to gain more understanding of the behaviour of these nanocrystals during a catalytic reaction. The particle size varied between 50 to 300 nm. Two preparation techniques have been used:

(1) The Pt THH synthesis and the alloying were performed using an electrochemical procedure at TU Eindhoven (Netherlands) according to Ref. [1]. Electrochemical surface alloying employed underpotential deposition of Cu from the respective precursor solutions.

(2) Pt NPs were also grown by thermal dewetting of Pt thin films on a sapphire substrate leading to an epitaxial relationship between the particle and the substrate (coll.: Pr. E. Rabkin of Technion-Israel).

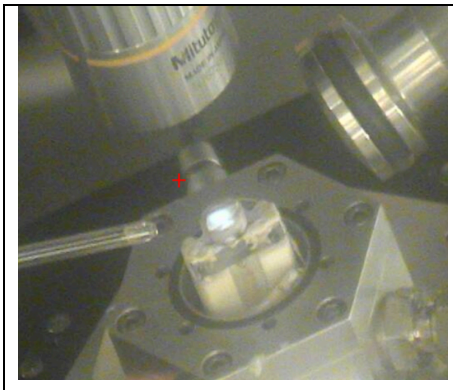

Figure 1: Photograph of the experimental set-up including the reactor (here, without dome), the vertical microscope and the focusing optics.

Selected single nanoparticles have been characterized by performing a 3D coherent x-ray diffraction map to give access to the shape and strain of the NP. The diffracted x-rays were detected using the 2D EIGER 1M pixel detector. Note that we were the first users to work with the new 2D EIGER detector at the ID01 beamline. We lost a lot of time debugging it. Bragg-CDI was performed under reaction conditions (*i.e.* under different gas atmospheres). CO oxidation was employed as test systems. We have developed an *in situ* reactor compatible with nano-focused x-ray beam [2]. The reactor allows to follow *in situ* the structural development of individual NPs with temperature and gas composition changes. In the future, we plan to add water-cooling to the furnace for better stability. Figure 1 displays a photograph of one sample glued on the heater of the reactor. The vertical microscope and the focusing optics (*i.e.* the order sorting aperture – OSA) are well observed. A mass spectrometer has been connected to the chamber to analyse the products of the reactions. We could not detect CO_2 due to the too large volume of the reactor.

Figure 2 displays reciprocal space maps around the **111** Pt reflection for an individual 200-nm diameter Pt particle at 450°C and under exposure to inert gas (Ar). Streaks coming from facets as well as thickness fringes are well observed

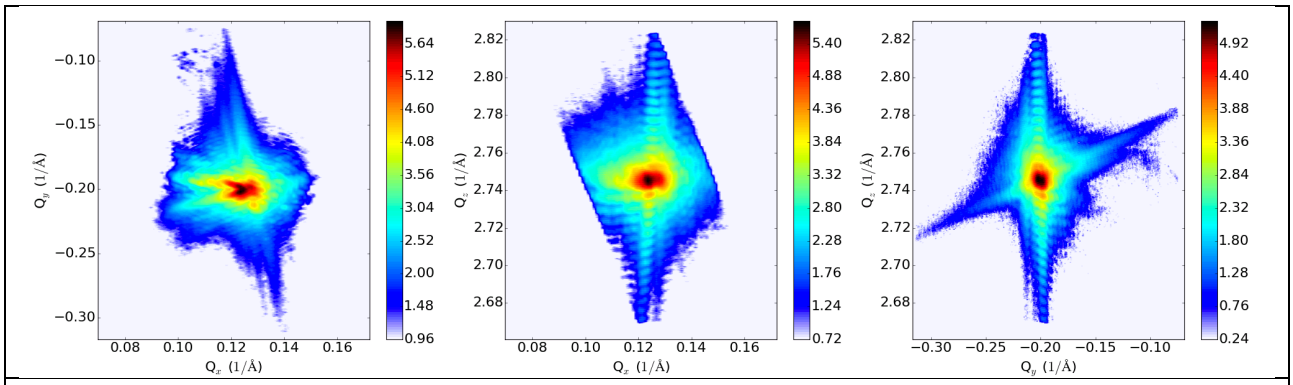

Figure 2: Reciprocal space maps around the **111** Pt reflection for an individual 200-nm diameter Pt particle at 450°C and under exposure to inert gas (Ar)

We were able to track the 3D diffraction pattern of an individual Pt particle at 450°C under different gas mixtures (see Figure 3). Streaks are observed to evolve while changing the gas mixtures. Figure 4 displays the reconstructed phase and density of the particle under CO. A twinned particle is reconstructed. Its phase is non-uniform; implying that the particle is strained by the substrate.

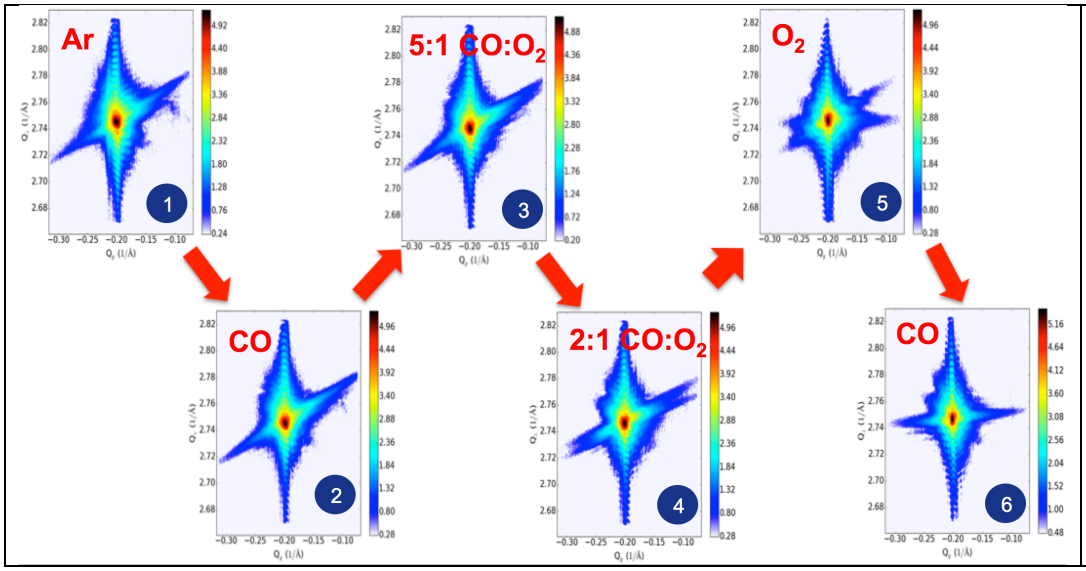

Figure 3: Reciprocal space maps around the **111** Pt reflection for an individual 200-nm diameter Pt particle at 450°C under exposure to different gas mixtures.

The proposed Bragg CDI study demonstrates the possibility to investigate the structure, strain and/or restructuring of catalyst particles under *in situ* conditions.

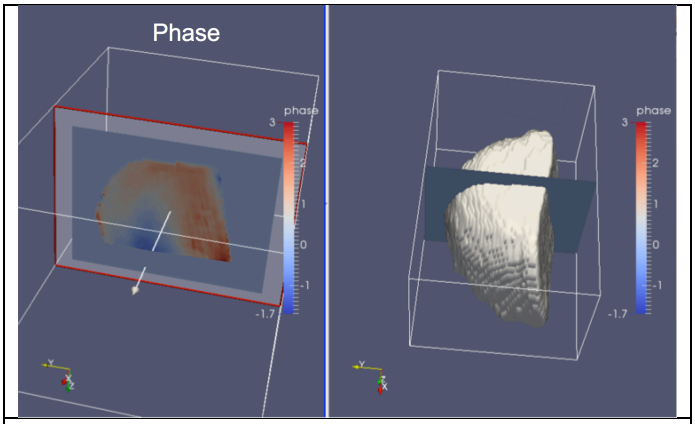

Figure 4: Reconstructed phase (left) and density (right) of the particle under CO.

References:

[1] N. Tian, Z.-Y. Zhou, S.-G. Sun, Y. Ding, and Z. L. Wang, Science **316**, 732 (2007).

[2] M.-I. Richard, S. Fernández, J. P. Hofmann, L. Gao, G. A. Chahine, S. J. Leake, H. Djazouli, Y. De Bortoli, L. Petit, P. Boesecke, S. Labat, E. J. M. Hensen, O. Thomas, and T. Schülli, Rev. Sci. Instrum. **88**, 093902 (2017).