	Experiment title: In-Situ Coherent X-Ray Diffraction Imaging of Surface Alloyed High-Index Faceted Platinum Particles	**Experiment number**: CH4760
Beamline: ID01	**Date of experiment**: from: February 22 2017 to: February 28 2017	**Date of report**: 27.03.2018
Shifts: 18	**Local contact(s)**: Dr. Marie-Ingrid Richard	*Received at ESRF:*

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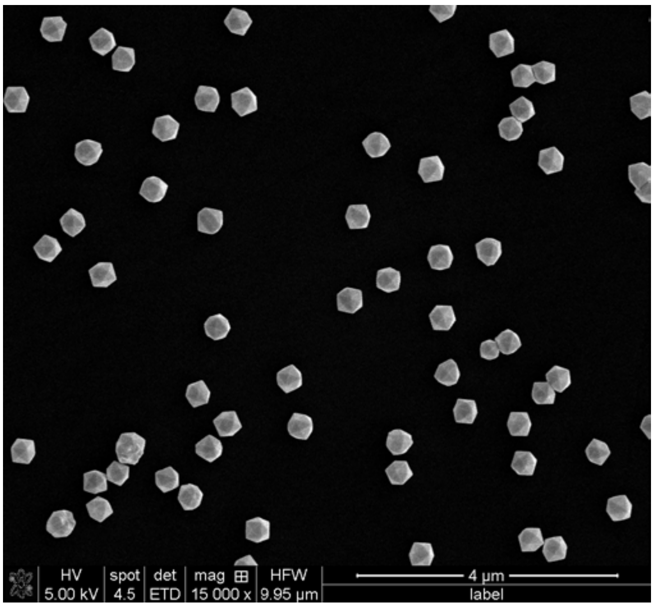
Report:

Our goal was to study the structural and catalytic properties of single Platinum (Pt) nanocrystals surface alloys using coherent X-ray diffraction imaging (CDI) in Bragg condition under *ex situ* and *in situ* electrochemical 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 surface alloy and de-alloy processes. High-index tetrahexaedral (THH) Pt nanocrystals (NCs) were electrochemically synthesized and then a monolayer of Cu was underpotentially deposited on the facets of Pt THH NCs. The sample was then annealed at 400 °C to make surface alloys (see Fig. 1). The diameter of the Pt NCs varied from 50 to 500 nm.



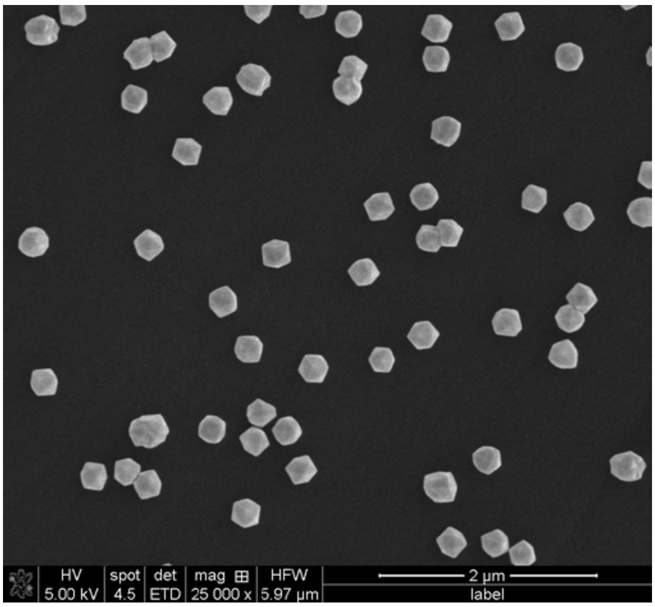


Fig. 1 *Left: Representative scanning electron microscopy (SEM) image of THH Pt nanocrystals. Right: SEM image of THH Pt/Cu surface alloy nanocrystals*

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 coherent beam was focused down to 700 nm×400 nm using a circular Fresnel Zone plate or Kirkpatrick-Baez mirrors. The nano-diffraction experiment was carried out at beam energy of 8 keV, so that the **002** Pt Bragg peak was accessible at a scattering angle of 23.269°. Isolated NCs have been located using the quicK-Mapping technique developed at beamline ID01. Three dimensional data sets were recorded by rocking the sample over a range of 2.6° in steps of 0.02°. We found bigger particles (>200 nm), which were stable under nano-focused X-ray beam and succeeded to measure a series of Pt and alloy NCs. 3D X-ray diffraction images were reduced into 2D stereographic projections with the center of sphere located at the center of mass of the 3D diffraction pattern. For the Pt THH, the best fit of the positions of the intense peaks resulted in a set of 12 poles of **201** type, corresponding to 12 {210} facets. For the Pt/Cu surface alloy, besides the **201**-type poles, a **205**-type pole was found, implying an extra facet of {250}.[1]

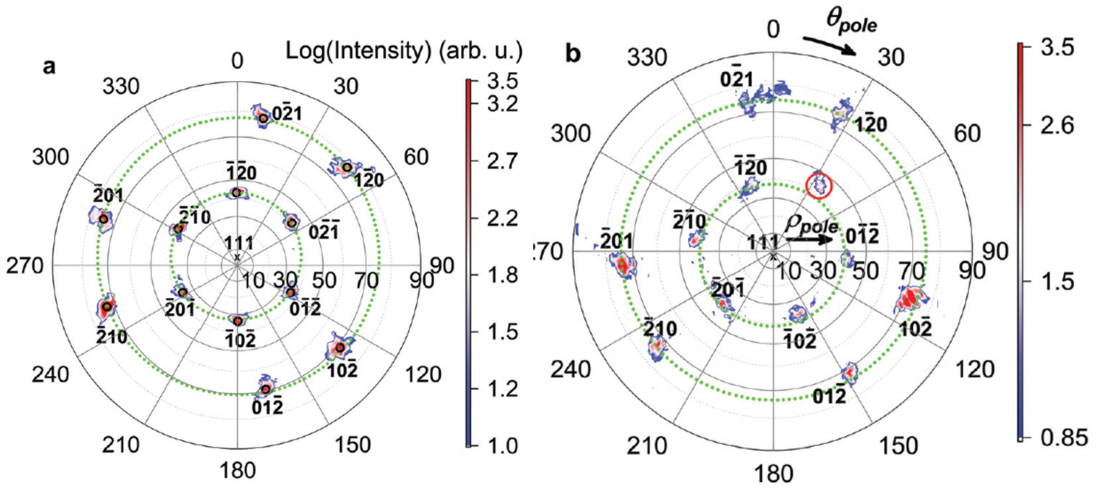


***Fig. 2 a)** Stereographic projections of the 3D reciprocal space map of a Pt THH particle. **b)** Stereographic projections of the 3D reciprocal space map of a Pt/Cu surface alloy particle.*

The representative isosurface strain distributions of Pt THH, Pt/Cu surface alloy and Pt/Cu surface alloy after etching are shown in Figure 3. The statistical analysis of the strain change after different treatment is still on the way.

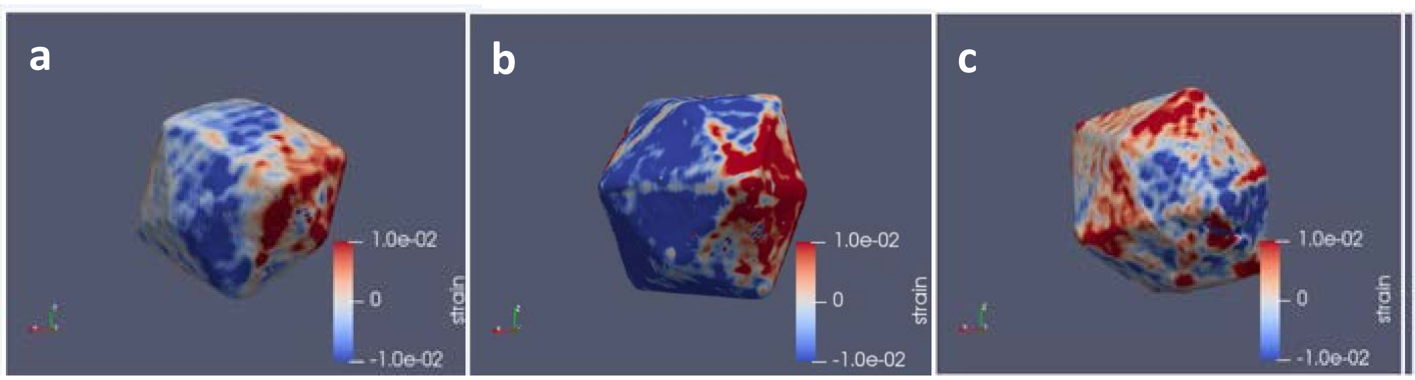


***Fig. 3** Isosurface strain distribution of **a)** a Pt THH particle, **b)** a Pt/Cu surface alloy particle, **c)** a Pt/Cu surface alloy after etching.*

The data and analysis of beamtime CH4760 have led to a publication in *Nanoscale* [1].

References:

[1] M.-I. Richard, S. Fernández, J. Eymery, J. P. Hofmann, L. Gao, J. Carnis, S. Labat, V. Favre-Nicolin, E. J. M. Hensen, O. Thomas, T. Schülli, S. Leake; *Crystallographic orientation of facets and planar defects elucidated by nano-focused coherent diffractive x-ray imaging; Nanoscale*, **10**, 4833-4840, **2018**.