



	Experiment title: <i>In Situ Coherent X-Ray Diffraction Imaging of High-Index Faceted Platinum Particles</i>	Experiment number: HC3796
Beamline:	Date of experiment: from: 29 oct. 2018 to: 02 nov. 2018	Date of report: 12/02/2019
Shifts:	Local contact(s): Marie-Ingrid Richard (marie-ingrid.richard@univ-amu.fr)	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Jerome Carnis ^{1,2,*} , Marie-Ingrid Richard ^{1,2,*} , Lu Gao ^{3,*} , Jan-Philip Hofmann ^{3,*} , Stephane Labat ¹ , Olivier Thomas ¹ , Eugene Rabkin ⁴ . ¹ Aix Marseille Université, CNRS, Université de Toulon, IM2NP UMR 7334, 13397, Marseille, France. ² ID01/ESRF, The European Synchrotron, CS 40220, F-38043 Grenoble Cedex 9, France. ³ Laboratory of Inorganic Materials Chemistry, Department of Chemical Engineering and Chemistry, Groene Loper 5, 5612 AE Eindhoven, The Netherlands. ⁴ Department of Materials Science and Engineering, Technion – Israel Institute of Technology, 32000 Haifa, Israel.		

Report:

During this experiment, we studied single Platinum nanoparticles (Pt) both ex-situ and in-situ applying Bragg coherent X-ray diffraction imaging and single-particle diffraction. These particles were synthesized by a new method allowing performing in-situ operando single particle structural studies during electrochemical reactions thanks to the conductive glassy carbon substrate. After checking the stability of particles ex-situ, we used the electrochemical cell developed by our collaborators at TU Eindhoven and a potentiostat borrowed from ESRF electrochemical laboratory. Our goal was to measure BCDI datasets at different potential values during the hydrogen evolution reaction and oxygen evolution reaction as test reactions (water splitting). We measured BCDI data for several crystals at open circuit potential and then measured the evolution of the center of mass of the Bragg peak for single particles during voltage steps, which teaches us about the evolution of lattice tilt and strain during surface processes. Finally, we performed preliminary tests to check the feasibility of following the electrochemical growth of a single PT nanoparticle using Bragg CDI. We investigated in details the best experimental procedure to measure nanoparticles in electrochemical solution while still mitigating X-ray induced effects.

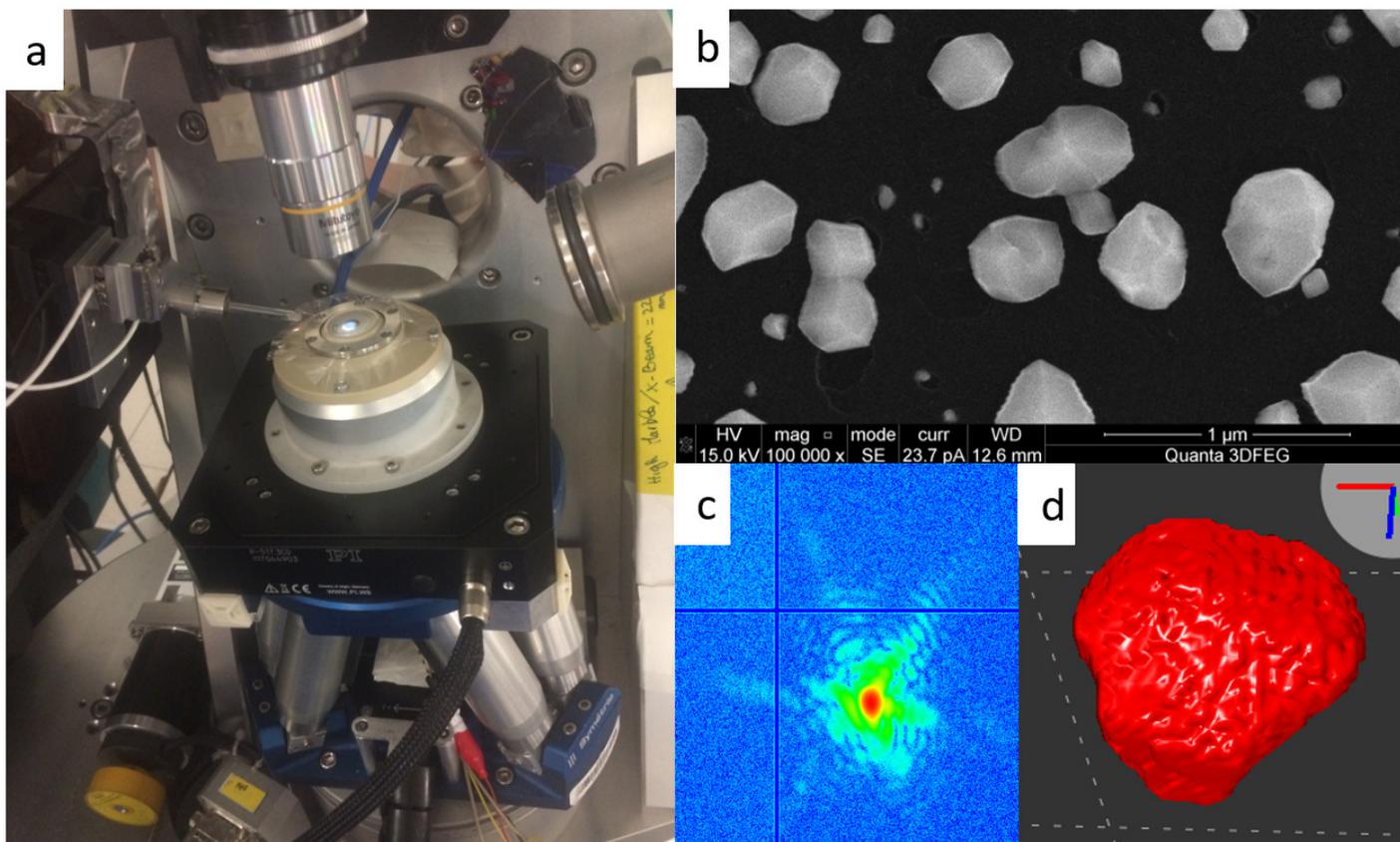


Figure 1: a) Our electrochemical cell mounted on ID01 diffractometer. b) High-index faceted Pt nanoparticles on a glassy carbon substrate, used during this experiment. c) Image of the diffraction pattern of a single Pt nanoparticle on ID01 Maxipix detector. d) Live reconstruction of the diffraction pattern in c), using PyNX software and SILX library.

Live data analysis is a critical point for this kind of experiments. We ran PyNX phase retrieval software using beamline computer to get a quick idea about the quality of the data we were measuring. The main difficulty came from the fact that X-rays interact strongly with the electrochemical solution, creating radicals, which can drive unwanted chemical reactions at the surface of nanoparticles. For potentials close to the oxygen evolution reaction, this resulted in breaking the link between the substrate and the particle illuminated by X-rays. In the presence of a photosensitive electrochemical solution, we observed the nucleation of smaller islands on existing Pt particles, leading to an uncontrolled growth. The experiment was therefore challenging, but we could identify trends in strain and tilt of single nanoparticles depending on the potential applied to the electrochemical cell, which directly links to the state of the surface (oxidized, metallic, with adsorbed species...).

We were not able to perform the originally proposed experiment of CO oxidation on Pt nanoparticles due to the absence of a carbonyl trap at the beamline, necessary to avoid Pt surface contamination by $\text{Ni}(\text{CO})_4$. However, we got promising results about the possibility of electrochemical studies based on BCDI. We were able to identify the main issues due to X-ray beam – electrolyte interactions and to design adapted experimental procedures.