EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Towards the observation of the behaviour of a single nanoparticle under catalytic reaction conditions	**Experiment number**: CH-4387
Beamline: ID16B-NA	**Date of experiment**: from: 22/4/2015 to: 28/4/2015	**Date of report**: 22/2/2017
Shifts: 15	**Local contact(s)**: Remi Tucoulou	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Andrew M. Beale* (UCL), David J. Martin* (UCL)

Report:

Precious metal nanoparticles comprising Pt, Au, Pd etc. supported on light metal oxides (i.e. Al_2O_3, SiO_2, TiO_2) form the basis of many types of industrial heterogeneous catalyst systems. It is well known that nanoparticles actively respond to their environment *in situ*, often changing shape so as to maximize, for example, the contact area of a particular surface. Cutting edge synthesis techniques such as reverse micelle metal ion encapsulation, first demonstrated by Spatz et al., generate nanoparticle populations with suitably small particle size distributions (σ << 1 nm), and critically, completely encapsulate all metal ions which prevents the development of sub-species by virtue of an water-in-oil system where solubility is fully controlled.

State of the art nano-beamlines such as the insertion device line ID16B-NA (ESRF) now possess the ability to perform conventional X-ray based techniques, such as XAS and XRD (X-ray diffraction), with extremely fine spatial resolution – with beam sizes in the range of 10s of nanometers. In these exeperiments we tested a new reactor cell and measured some supported Pd catalysts under a controlled atmosphere (see Figure 1). XAFS data were collected on two samples (Si_3N_4 window located on the reactor cell window) at 293 K under He flow, and under different atmospheres at 343 K (H_2, O_2, CO and a reactive mixture of CO and O_2) using a 400 × 400 nm^2 square beam. Figure 2 shows XANES spectra of a sample as well as XANES spectra from a Pd foil. The data obtained for sample A-1 is noisy as the spectra is only collected from ca. 150 particles, however, it is clear that there are several characteristic “peaks” (continuum resonances and scattering features) primarily located at 24.36, 24.38 and 24.42 keV respectively, ascribable to metallic Pd.

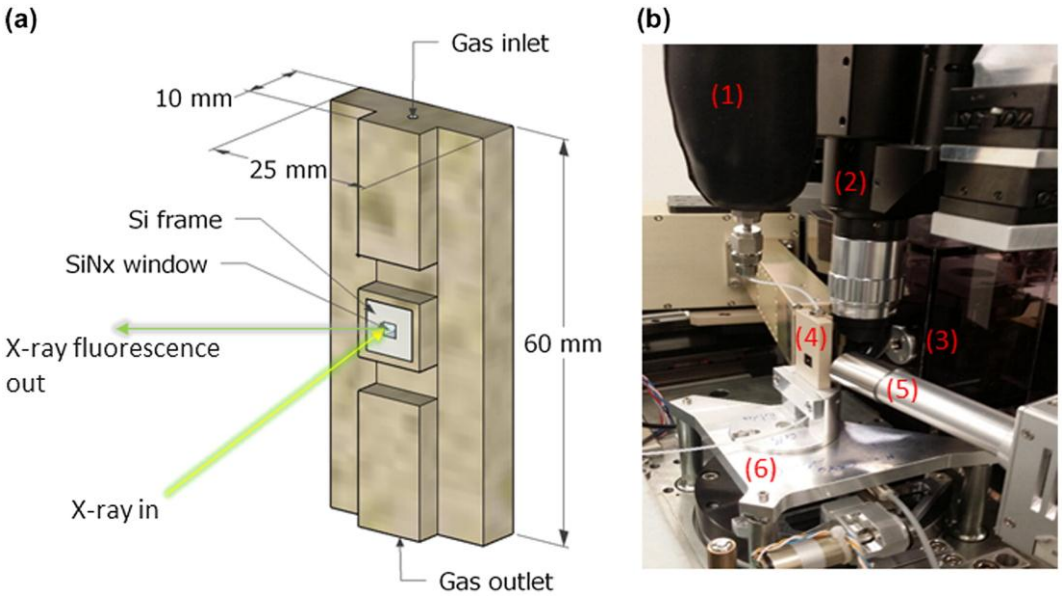

Figure 1. (a) Schematic of the gas flow cell used on ID16B-NA. Gas flows from top to bottom, across the SiN_xwindow which is loaded with Pd particles. (b) Close up of the reactor set up. The gas flows in from the heated tubing at the top, passes across the Pd-SiN_x window and out through the bottom to exhaust. Two XRF detectors (single and multi-element array) are positioned extremely close to the front-side window at ca. 15° to collect as high quality data as possible.

In summary we obtained uniform Pd nanoparticles using a reverse micelle synthesis method and demonstrated that it was possible to obtain spectroscopic information using a nanosized X-ray beam from as few as ~20 nanoparticles (we estimate) allowing us to analyse nanoparticle behaviour under various reactive gas atmospheres at fixed temperatures. These preliminary data presented here represents a first attempt to examine single nanoparticles, which could help remove statistical averaging effects that accompanies bulk XAS techniques.

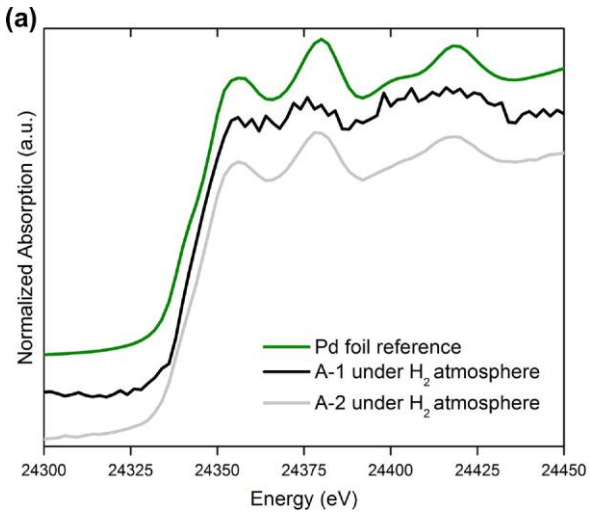

Figure 2. (a) Normalized XANES spectra of sample A-1 and A-2 against a palladium foil reference.