EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

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: In situ XAFS investigation of the electronic and structural properties of novel model catalysts based on deposited size-selected Au/Pt bimetallic gas phase clusters**	**Experiment number**: **26-01-970**
Beamline: BM-26A	**Date of experiment**: from: 07 Jun 2013 to: 11 Jun 2013	**Date of report**: 10/10/2013
Shifts: 12	**Local contact(s)**: Dipanjan Banerjee & Sergey Nikitenko	*Received at ESRF:*

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Report:

In the framework of a multidisciplinary project we are developping a new approach to produce novel catalysts based on size-selected mono and bimetallic gas-phase clusters deposited on various substrates. Contrarily to the classical chemical method this production method can synthesize series of supported metal catalysts with a controlled and highly reproducible homogeneity using a cluster deposition set-up based on a laser vaporization source operating under UHV conditions.[1,2]

One of the typical limitation of this technique is the small amount of clusters that can be deposited onto surfaces in comparison to volumes for chemically prepared nanoparticles. To obtain a cluster loading sufficient (ca. 1 wt %) to allow XAFS measurements in fluorescence mode and catalysis activity monitoring, we have deposited metallic clusters on both sides of ultrathin (few μm) porous borosilicate supports that are subsequently ground into powder. Our recent experience has shown that the amount of clusters corresponding to several cm^2 of double-sided cluster deposition was sufficient to produce a detectable amount of CO_2 from the CO oxidation by O_2 in a laboratory flow bed reactor.

For this experiment we have prepared size-selected mono and bi-metallic Au and Pt-based gas phase clusters (ca. 2.5 nm), deposited with low coverage (0.5-1 ML -MonoLayer-) to prevent agglomeration onto borosilicates. The planned experiment was to measured these samples in situ in capillaries under various gas flows and temperatures using the integrated gas rig system recently installed at Dubble to mimic the laboratory CO oxidation conditions. The temperature provided with a heater gun was to be raised to 550ºC at

a rate of 5 ºC / with 4 plateaus at RT, 150, 450 and 550 ºC programmed for combined XANES and EXAFS measurements.

Although Dubble gas rig integrated system has proved in our previous experiments to be efficient and flexible we have unfortunately run this time into series of serious technical and safety issues. This has forced us after many unsuccesful time consuming attempts (3-4 shifts) to finally cancel all the gas treatments and *in situ* measurements that were orignally planned in our proposal.

In the remaining time we have been able to successfully measure XAFS of our mono and bimetallic samples in air and at room temperature at Au and Pt L_3 edges in fluorescent mode using the Ge 9-channel detector. Collection of up to 20 scans with typical acquisition times of 30 min up to k = 10 $Å^{-1}$ was required to obtain an acceptable signal to noise ratio for the bimetallic clusters that feature the lowest Pt loading. The averaged Pt-L_3 edge absorption spectrum of the AuPt bimetallic sample is presented in Figure 1. Despite the apparent limited data quality, crucial information such as the metal-metal distances, oxide content, and metal-support interactions needed for the extensive structural characterisation of these bimetallic metal clusters could be obtained with a detailed XAFS analysis.

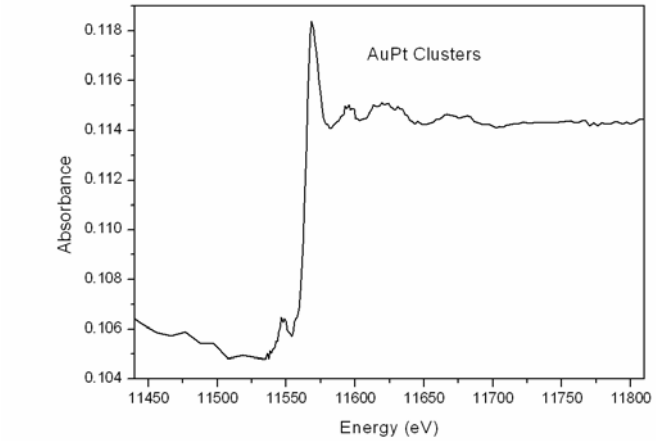

Figure 1: Averaged Pt-L_3 edge X-ray absorption spectrum of AuPt bimetallic clusters deposited on borosilicate (pre-edge feature is a tungsten impurity orignating from the substrate machining).

We are now ready to resubmit our proposal to perform the *in situ* measurements once Dubble gas rig is fully operational again.

(1)Bouwen, W.; Thoen, P.; Vanhoutte, F.; Bouckaert, S.; Despa, F.; Weidele, H.; Silverans, R. E.; Lievens, P. *Rev. Sci. Instr.* **2000**, *71*, 54.
(2)Neukermans, S.; Wang, X.; Veldeman, N.; Janssens, E.; Silverans, R. E.; Lievens, P. *J. Mass Spectr.* **2006**, *252*, 145.