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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** Operando XAS study of Cu-Ga nanoparticles as catalysts for electrochemical CO_2 reduction	**Experiment number**: A31-1-182
Beamline: BM31	**Date of experiment**: from: 02.10.22 to: 08.10.22	**Date of report**: 07.09.23
Shifts: 14	**Local contact(s)**: Dragos Stoian	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
Valery Okatenko[1] *
Dr. Mark A. Newton[1] *
Petru Albertini[1] *
Dr. Dragos Stoian[2] *
Prof. Raffaella Buonsanti[1]

[1] Laboratory of Nanochemistry for Energy Research, Institute of Chemical Sciences and Engineering, Ecole Politechnique Fédérale de Lausanne, Sion, CH-1950, Switzerland
[2] Swiss-Norwegian Beamlines, European Synchrotron Radiation Facility, 38000 Grenoble, France

Report:

Hybrid organic/inorganic materials have contributed to solve many challenges in different areas of science.[1–5] One of the biggest challenges for a more sustainable society is to have active and stable catalysts which enable the transition from fossil to renewable feedstocks, reduce energy consumption, and minimize the environmental footprint. Herein, we synthesize novel hybrid materials where an amorphous oxide coating with embedded organic ligands surrounds metallic nanocrystals. We demonstrate that the hybrid coating is a powerful mean to create electrocatalysts stable against structural reconstruction during the CO_2 electroreduction. These electrocatalysts consists of copper nanocrystals encapsulated in a hybrid organic/inorganic alumina shell. This shell locks a fraction of the copper surface into a reduction resistant Cu^{2+} state, which inhibits those redox processes responsible for the structural reconstruction of copper. The electrocatalyst activity is preserved, which would not be possible with a conventional dense alumina coating. Varying the shell thickness and the coating morphology yields fundamental insights into the stabilization mechanism and emphasizes the importance of the Lewis acidity of the shell in relation to the retention of catalyst structure. The synthetic tunability of the chemistry developed herein opens new avenues for the design of stable electrocatalysts and beyond.

The preprint of this work is available as:

Petru P. Albertini, Mark A. Newton, Min Wang, Ona Segura Lecina, Philippe B. Green, Dragos C. Stoian, Emad Oveisi, Anna Loiudice, Raffaella Buonsanti. Hybrid Oxide Coatings Generate Stable Cu Catalysts for CO_2 Electroreduction. DOI: 10.21203/rs.3.rs-2873572/v1.

The paper is submitted to Nature Materials and is currently under revision.