EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

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 insights on CuGa nanoparticles in electrochemical CO2 reduction	**Experiment number**: A31-1-145
Beamline: BM31	**Date of experiment**: from: 14.02.22 to: 18.02.22	**Date of report**: 21.02.23
Shifts: 9	**Local contact(s)**: Dragos Stoian	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Valery Okatenko*

Raffaella Buonsanti

Dragos Stoian

Report:

Copper nanocatalysts are among the most promising candidates to drive the electrochemical CO_2 reduction reaction (CO_2RR). However, the stability of such catalysts during operation is sub-optimal, and improving this aspect of catalyst behavior remains a challenge. Here, we synthesize well-defined and tunable CuGa nanoparticles (NPs) and demonstrate that alloying Cu with Ga considerably improves the stability of the nanocatalysts. In particular, we discover that CuGa NPs containing 17 at. % of Ga preserve most of their CO_2RR activity for at least 20 hours while Cu NPs of the same size reconstruct and lose their CO_2RR activity within 2 hours. Various characterization techniques, including X-Ray photoelectron spectroscopy and *operando* X-Ray absorption spectroscopy, suggest that the addition of Ga suppresses Cu oxidation at open circuit potential (ocp), and induces significant electronic interactions between Ga and Cu. Thus, we explain the observed stabilization of the Cu by Ga as a result of the higher oxophilicity and lower electronegativity of Ga, which reduce the propensity of Cu to oxidize at ocp and enhance the bond strength in the alloyed nanocatalysts. In addition to addressing one of the major challenges in CO_2RR, this study proposes a strategy to generate NPs which are stable under a reducing reaction environment.

The results are published in *J. Am. Chem. Soc.* with DOI: 10.1021/jacs.2c13437.