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:** Structural transformation of core-shell CoNi nanoparticles during magnetic catalysis	**Experiment number**: CH-6055
Beamline: BM25,Spline	**Date of experiment**: from: 15th September 2021 to: 21th September 2021	**Date of report**: 15/06/2022
Shifts: 18	**Local contact(s)**: Juan Rubio	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The main objective of this study was to gain information about the structural evolution of core-shell Cobalt-Nickel nanoparticles (**Co@Ni@C**) as well as identify the nature of the active sites under reaction conditions (CO_2 hydrogenation). For this, we followed a two-step strategy: i) the realization of ex-situ high-resolution XRD on **Co@Ni@C** at different reaction stages. The reactions performed in the laboratory were interrupted at different process stages. Thus, five samples were measured, corresponding to the initial (before catalysis), final (after catalysis) and three intermediate catalysts (28, 32 and 41 mT) and analysed under standard conditions (room temperature and air). (**Figure 1**). ii) In the second strategy (in-situ) the catalyst was placed in a vacuum chamber, heated at different temperatures (25-700 ºC) and brought into contact with the reactant gases (CO_2 and H_2). The reactants were transported in a He flow, pressure total 1 bar and the H_2 and CO_2 concentration below 10% (**Figure 2**).

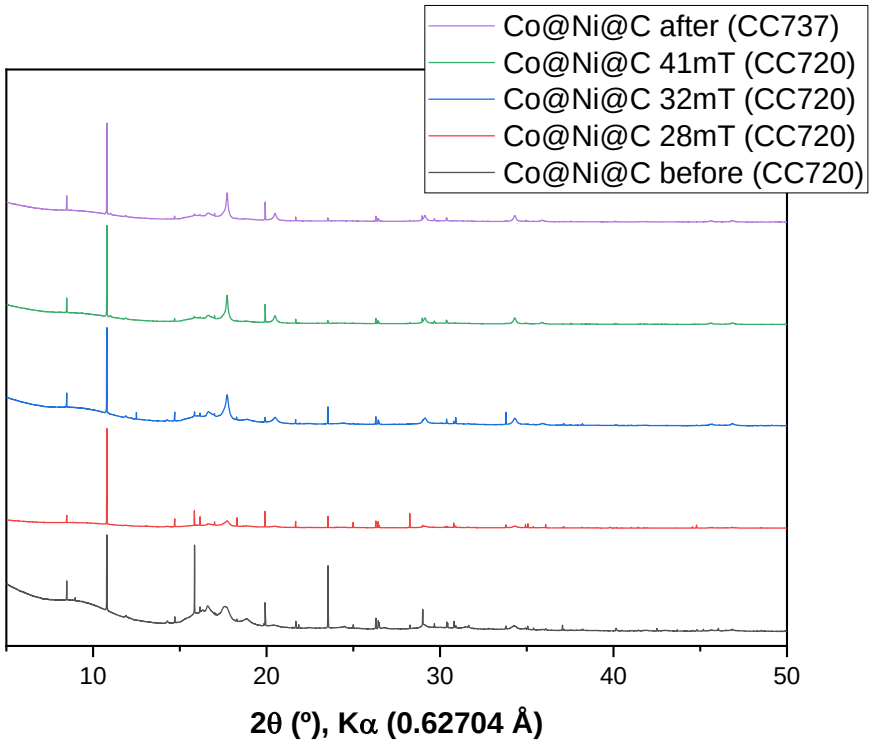

Figure 1. Ex-situ high-resolution XRD on **Co@Ni@C** at different reaction stages.

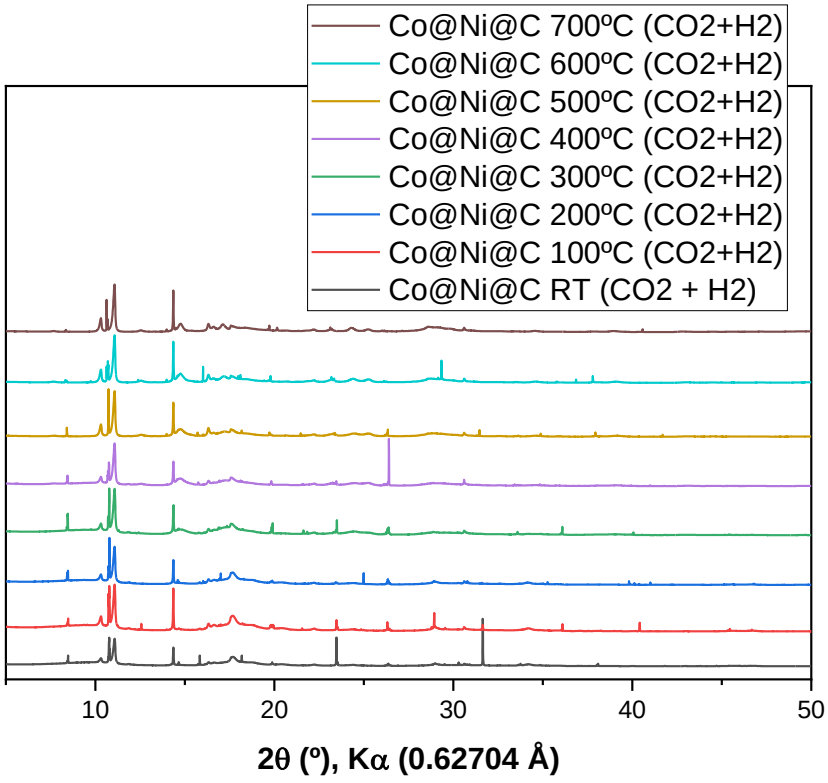

Figure 2. In-situ high-resolution XRD on **Co@Ni@C** at different temperatures (25-700 ºC) in flow conditions (CO_2 and H2).

High-resolution XRD experiments indicates that Co@Ni@C undergoes crystallographic changes, which may alter the catalytic activity under the magnetically induced catalysis, evidencing a structural transformation. However, detailed analysis is necessary to identify this structural change.