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: "Investigation of the nanostructure development of a PEG-polyacrylate during UV curing"**	**Experiment number**: MA-4398
Beamline: BM26B	**Date of experiment**: from: September 10th 2018 to: September 14th 2018	**Date of report**: Sep. 24th 2019
Shifts: 9	**Local contact(s)**: Dr. Michela Brunelli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Bing Wu* – Dubble/Royal College of Surgeons in Ireland

Daniel Hermida Merino – Dubble/ NWO

Report:

Supercapacitors have been considered as promising energy-storage devices with advantages that include increasingly fast charge–discharge rates, higher power density, longer cycle stability, and potentially low cost. The first reported low internal resistance supercapacitors, electric double-layer capacitors (EDLCs), were made of carbonaceous materials in which the charge storage was predominantly achieved by double-layer capacitance of physical separation of charges. The simplicity and high stability of EDLCs made them very attractive. However, with capacitance only in the range of 100–300 F/g, they do not meet the requirements for modern energy-storage devices. More recently pseudocapacitors, in which charge is primarily stored through reversible electrochemical processes (redox, electro-sorption and/or intercalation) on the electrode surface, have attracted attention due to their intrinsically higher capacitance.

Currently we are developing a simple, scalable and novel approach to produce super-capacitive high graphene content materials that uses a biopolymer to prepare 3D carbon nanocomposite doped with highly dispersed Co-Ni nanoparticles. Persimmon tannin and chitosan were employed as the carbon source. Persimmon tannin contains a number of adjacent hydroxyphenyl groups, which are suitable ligands for transition-metal ions such as Ni and Co ions. During the carbonization process, the complexes were decomposed into metal nanoparticles doped carbon. Meanwhile, metal nanoparticles catalyzed the formation of 3D graphene. The materials produced from this approach were well characterized through different spectroscopic techniques, and the electro-chemical properties of this material also shows great improvement in its supercapacities.

By analysing this system with synchrotron-based operando CV-SAXS experiment the size and distribution of these embedded nanoparticle in relation to the electrochemical performance of this nanocomposite can be further understood at

the nano-scopic level. On the other hand, the impact of charging-discharging on the microstructure of this nanocomposite material can be better illustrated through the in-situ SAXS-WAXS analyses.

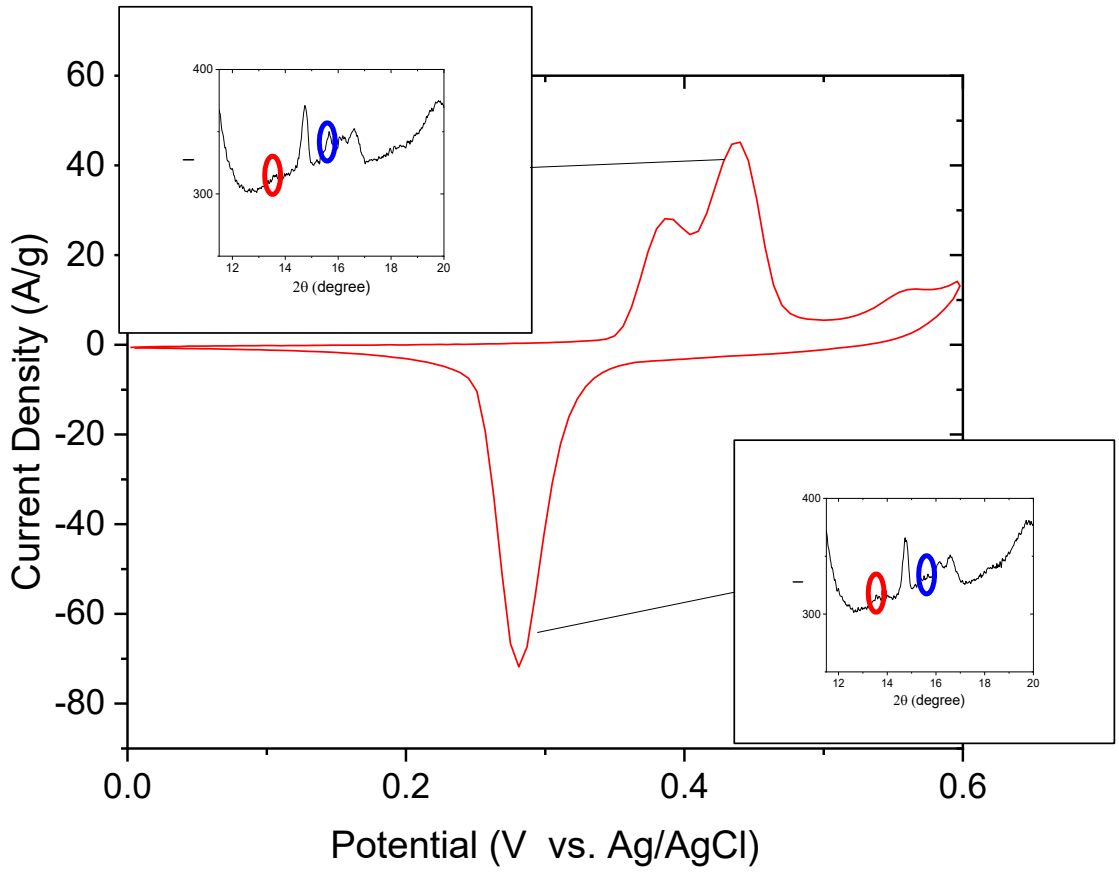

Figure 1. Charge-discharging profile of G-CoO-NiO nanocomposite. Insert: WAXS profiles at the redox peaks during he charging-discharing period.

As shown in the Figure 1, the application of voltage on the G-CoO-NiO really changed the sample's WAXS pattern at diffraction angle around 14 and 16 degree. These peaks can be found correspondign to CoO peaks, this turn demonstrate the redox reaction happened during the charge-discharge cycle is predominantly a CoO to Co based reaction. Further study is needed to understand the role of Ni in this reaction. On the other hand, it seems that there is no change in the SAXS profiles for this redox reaction, which indicates a good dispersion of NP in the carbon supports, and no aggregation appeared during redox reactions.

Next step, we plan to use the same setup to study a series of different nanocomposite to see the impact of its morphology on its electrochemical performance.