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

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:** Effect of the existence of metal vacancies in sodium layered oxide $Na_xFe_{1-y}Mn_yO_2$ phases used in sodium batteries on their electrochemical performances	**Experiment number**: CH-3877
Beamline: ID31	**Date of experiment**: from: 13/11/2013 to: 15/11/2013	**Date of report**: 15/12/2017
Shifts: 6	**Local contact(s)**: Christina Drathen	*Received at ESRF:* 15/12/2017

Names and affiliations of applicants (* indicates experimentalists):

Dr Claude Delmas

Dr Marie Guignard*

Benoit Mortemard de Boisse*

Dr Dany Carlier

Report:

This successful ID31 experiment allowed to determine the structure of a series of sodium layered oxides with the general formula $Na_xFe_{1-y}Mn_yO_2$ which were used as positive electrode in sodium batteries.

For $Na_{0.96}Mn_{1/3}Fe_{2/3}O_2$ and $NaMn_{1/2}Fe_{1/2}O_2$, we found from the atomic positions that the MO_6 octahedra (M = Mn and Fe) show two long M–O bonds and 4 shorter ones confirming the cooperative Jahn–Teller effect. It was surprising to found that only a small amount of Mn^{3+} ions (1/3 of the transition metal ions in $Na_{0.96}Mn_{1/3}Fe_{2/3}O_2$) was enough to generate a cooperative distortion of the structure.

As part of the complete composition-structure-property understanding for these materials, the ESRF data were combined with electrochemical measurements and Mössbauer sepctroscopy performed at the ICMCB. These results were used in 2 separate publications. Please refer to the publications listed below for details (and figures) of the results.

We thank Christina Drathen for providing assistance in using beamline ID31.

Publications incorpationing ESRF, ID31 powder XRD data

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B. Mortemard de Boisse, D. Carlier, M. Guignard, L. Bourgeois, C. Delmas "*P2-$Na_xMn_{1/2}Fe_{1/2}O_2$ Phase Used as Positive Electrode in Na Batteries: Structural Changes Induced by the Electrochemical (De)intercalation Process"* Inorganic Chemsitry 53 (2014) 11197-11205.

B. Mortemard de Boisse, J.-H. Cheng, D. Carlier, M. Guignard, C.-J. Pan, S. Bordère, D. Filimonov, C. Drathen, E. Suard, B.-J. Hwang, A. Wattiaux, C. Delmas *"O3-$Na_xMn_{1/3}Fe_{2/3}O_2$ as a positive electrode material for Na-ion batteries: structural evolutions and redox mechanisms upon Na^+ (de)intercalation"* Journal of Materials Chemistry A 3 (2015) 10976-10989.

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