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: Optimising the performance of bio inspired engineering materials**	**Experiment number**:
Beamline: ID15A	**Date of experiment**: from: 16/10/2018 to: 19/10/2018	**Date of report**: 12/09/2022
Shifts: 9	**Local contact(s)**: Samuel McDonald	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Samuel McDonald* [1]
Dr Shelley Rawson* [1]
Professor Philip J Withers [1]
Dr Suelen Barg [2]

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Report:

1. **Published article**

Bayram, V., Ghidiu, M., Byun, J.J., Rawson, S.D., Yang, P., Mcdonald, S.A., Lindley, M., Fairclough, S., Haigh, S.J., Withers, P.J. and Barsoum, M.W., 2019. MXene Tunable Lamellae Architectures for Supercapacitor Electrodes. ACS Applied Energy Materials, 3(1), pp.411-422. 2019.

Abstract:
The rich elemental composition, surface chemistry, and outstanding electrical conductivity of MXenes make them a promising class of two-dimensional (2D) materials for electrochemical energy storage. To translate these properties into high performance devices, it is essential to develop fabrication strategies that allow MXenes to be assembled into electrodes with tunable architectures and investigate the effect of their pore structure on the capacitive performance. Here, we report on the fabrication of MXene aerogels with highly ordered lamellar structures by unidirectional freeze-casting of additive-free $Ti_3C_2T_x$ aqueous suspensions. These structures can be subsequently processed into practical supercapacitor electrode films by pressing or calendering steps. This versatile processing route allows a wide control of film thickness, spacing within lamellae (to give electrolyte accessible sites), and densities (over 2 orders of magnitude) and hence gives control over the final properties. The as-prepared MXene aerogel with a density of 13 mg cm^{-3} achieves 380 F g^{-1} capacitance at 2 mV s^{-1} and 75 F g^{-1} at 50 mV s^{-1}. The calendering of the MXene aerogel into a porous 60 μm thick film with a density of 434 mg cm^{-3} leads to a superior rate capability of 309 F g^{-1} at 50 mV s^{-1}. In addition, the rolled electrodes present an improvement in volumetric capacitance of 104 times as compared to the as-prepared MXene aerogel. Finally, the outstanding cyclability of rolled electrodes strengthens their nomination for supercapacitor applications. In this paper we demonstrate the possibilities in tuning the porosity and the electrochemical properties of aerogels highlighting the importance of evaluating new and hybrid processing methods to develop energy storage applications. The simplicity and versatility of the developed fabrication strategy open

opportunities for the utilization of MXene lamellae architectures in a wide range of applications requiring controlled porosity including catalysis, filtration, and water purification.

2. Published article

Rawson, S.D., Bayram, V., McDonald, S.A., Yang, P., Courtois, L., Guo, Y., Xu, J., Burnett, T.L., Barg, S. and Withers, P.J. Tailoring the Microstructure of Lamellar $Ti_3C_2T_x$ MXene Aerogel by Compressive Straining. ACS Nano, 16(2), pp.1896-1908. 2022.

Abstract:
Aerogels are attracting increasing interest due to their functional properties, such as lightweight and high porosity, which make them promising materials for energy storage and advanced composites. Compressive deformation allows the nano- and microstructure of lamellar freeze-cast aerogels to be tailored toward the aforementioned applications, where a 3D nanostructure of closely spaced, aligned sheets is desired. Quantitatively characterizing their microstructural evolution during compression is needed to allow optimization of manufacturing, understand in-service structural changes, and determine how aerogel structure relates to functional properties. Herein we have developed methods to quantitatively analyze lamellar aerogel domains, sheet spacing, and sheet orientation in 3D and to track their evolution as a function of increasing compression through synchrotron phase contrast X-ray microcomputed tomography (μCT). The as-cast domains are predominantly aligned with the freezing direction with random orientation in the orthogonal plane. Generally the sheets rotate toward flat and their spacing narrows progressively with increasing compression with negligible lateral strain (zero Poisson's ratio). This is with the exception of sheets close to parallel with the loading direction (*Z*), which maintain their orientation and sheet spacing until ~60% compression, beyond which they exhibit buckling. These data suggest that a single-domain, fully aligned as-cast aerogel is not necessary to produce a post-compression aligned lamellar structure and indicate how the spacing can be tailored as a function of compressive strain. The analysis methods presented herein are applicable to optimizing freeze-casting process and quantifying lamellar microdomain structures generally.