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 fracture toughness through tailored multi-scale microstructure	**Experiment number**:
Beamline: ID19	**Date of experiment**: from: 10/9/2021 to: 13/9/2021	**Date of report**: 12/09/2022
Shifts: 9	**Local contact(s)**: Alexander Rack	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Shelley D Rawson[1]*
Suelen Barg[2]
Florian Bouville[3]*
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Report:

1. Publication in preparation

Fracture toughening mechanisms in biomimetic Ti3C2Tx MXene aerogel-epoxy composites.
Shelley D Rawson, Pei Yang, Victoria Vilchez, Jamie McGregor, Matthew Lawson, Alexander Rack, Yunhui Chen, Katie Lewthwaite, Tian Xia, Florian Bouville, Suelen Barg, Philip J Withers.
Intended journal: Science and Technology of Advanced Materials
Intended submision date: 01/11/2022.

Abstract:
Programming good toughness into otherwise brittle materials is desirable to increase component life span or reduce weight. This combination of material properties can be achieved by incorporating nanostructure within the material, as achieved in freeze-cast MXene-epoxy aerogel composites. Inspired by nacre (mother-of-pearl), freeze-cast $Ti_3C_2T_x$ MXene aerogel-epoxy composites have a bricks-and-mortar-like nanostructure, thought to facilitate multi-scale toughening mechanisms. In order to investigate whether nacre-like toughening mechanisms occur, we have used synchrotron X-ray micro computed tomography (µCT) acquired during fracture to visualise the crack as it propagates through the MXene-epoxy aerogel composites nanostructure. The nanostructure gives rise to nacre-like fracture propagation behaviour including crack deviation and bifurcation, platelet bridging, a process zone ahead of the crack, and a nacre-like fracture surface. These mechanisms act to increase the work required to propagate a crack, provding increased fracture toughness to the material.

2. Publication in preparation

Influence of the secondary phase composition on the fracture response of nacre-like alumina.Victoria Vilchez, Shelley D Rawson, Jamie McGregor, Matthew Lawson, Yunhui Chen, Alexander Rack, Philip J Withers, Florian Bouville.
Intended Journal: Journal of the European Ceramic Society
Intended submission date: 01/01/2023

Abstract:
The high strength of ceramic materials is attractive across a range of demanding environments from energy generation to construction, however, the inherent brittleness of traditional ceramics has limited their application. The combination of high stiffness and high fracture toughness has been elusive in engineering; however, nature has provided several examples, such as nacre (mother of pearl) which remains the gold standard for high stiffness and toughness. Nacre comprises of a bricks and mortar architecture of ceramic platelets separated by an organic polymer, and this material has provided inspiration for nanopatterning of ceramics to increase their fracture toughness. The aim of this reaesrch was to compare nanopatterned ceramics of different composition and manufacturing parameters to determine their differing fracture behaviours. Magnetically assisted slip casting has been used to produce a nanopattened ceramics of alumina with a nacre-like architecture or alumina platelets separated by aluminium borate, yttria or Silica. Samples were sintered at temperature of 1350ºC or 1400ºC, producing four different compositions of nanopatterned alumina composites. Samples were then fractured by single edge notched bending *in situ* within the ID19 beamline of the Eurpoean Synchrotron Radiation Facility (ESRF) to allow observation of the evolution of the crack path during fracture. Results demonstrate that the crack length is not the same through the sampe thickness, highlighting the need for 3D data over 2D scanning electron microcopy of the sample surface. Alumina-aluminium borate samples exhibited greater propensity for lateral cracking as compared with aluminium-yttria and aluminium-silica samples. These findings provide guidance towards further development of magnetically assisted slip cast materials towards increased toughness.