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

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: Characterizing the 3D hierarchical structure of human enamel at the nanoscale**	**Experiment number**: MD-905
Beamline:	**Date of experiment**: from: 17/02/2016 to: 23/02/2016	**Date of report**: 22/03/2016
Shifts:	**Local contact(s)**: Steven LEAKE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Dental enamel is a unique hierarchical material. The aim of this experiment was to visualize the mosaic 3D crystalline organisation of individual enamel nanorods (namely, the crystalline coherence, strain field and lattice rotation), using 3D Bragg nanodiffraction and ptychography. These results are fundamental in understanding the structural and mechanical properties of enamel and may further guide biomimetic models for synthetic reparative dentistry.

This has been a successful experiment in several aspects:

1. **We have combined coherent diffraction imaging and nanodiffraction to obtain data that will allow us to characterise human enamel at the nanometre scale.**
2. **Diffraction data acquired from the (002) reflection (i.e., a reflection that is orthogonal to the c-axis of the hexagonal hydroxyapatite nanorods in enamel) using a fully coherent beam show interference fringes that with preliminary analysis we potentially ascribe (i.e. prior to full data analysis) to the enamel nanorods having a mosaic structure, instead of they being single crystals, as is generally assumed.**
3. **Due to the complexity of this mosaic structure that we have found, as a first instance we resorted to CDI instead to ptychography to study the individual small crystals within the larger nanorods.**

4. The coherent diffraction data are of very good quality. We have obtained several sets of data of isolated small crystals. Thus, it should be possible to use phase retrieval techniques to determine the shape of the small units that form the mosaic structure of the nanorods and their strain distribution.

5. In the scattering volume probed, reflections with only the first Miller index being non-zero showed that the nanorods have a random (or at least not well defined) rotational orientation, where the rotation axis is pointing along the long direction of the rods (the c-axis).

6. Reciprocal space maps around the (002) Bragg reflection, using a partial coherent beam, have been acquired at different regions of interest on the sample and should allow us to evaluate an average strain distribution in these areas. Maps have been acquired in 4 μm x 4 μm areas at different points along a line that goes from the enamel-dentine junction to the tooth surface through enamel to ascertain the differences in texture between the tooth inner and outer regions. In particular, we have observed a marked difference between enamel and dentine.

7. In addition, using several test samples (patterned motives on silicon, enamel) and observing the evolution of speckles formed by a grainy crystalline structure, we have determined that the setup of ID01 during our experiment was not stable enough for a full ptychography study at the nanoscale. For example, we have estimated a mean drift of ~ 80 nm/h along the vertical direction. The origin of this drift is uncertain.

Despite the drift issues that have precluded us from using a ptychographic approach, we would like to note that the beamline is very well optimised, especially for the strain measurements and raster maps we have carried out. We would also like to highglight the technical support of our local contact, Dr. Steven Leake, who has been extremely helpful and has given us perfect assistance when needed.

Addendum: More recent experiments at ID01 (HC-2282) have shown that 3D Bragg ptychography was possible so we are confident that such an experiment should be fully successful in the near future.