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:** High-resolution X-ray scattering tensor tomography study of the age-related structural changes in the human femoral neck cortex	**Experiment number**: SC5408
Beamline: ID13	**Date of experiment**: from: 12.07.23 to: 16.07.23	**Date of report**: 08.09.23
Shifts: 12	**Local contact(s)**: Manfred Burghammer	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Overview

This experiment is part of a broader investigation aiming to assess the contribution of bone tissue properties to the strength of the ageing human hip. The aim of this experiment was to analyse the orientation and spatial distribution of the collagen and mineral platelets in the human femoral neck cortex at the lamellar level, in the aim of relating the structure to the mechanical properties. A total of 4 micropillars, extracted from the inferior and superior femoral neck cortex of 2 patients and previously tested by microcompression and Raman spectroscopy, were measured by small- and wide-angle X-ray scattering tensor tomography for structural (SWASTT). Additionally, a separate absorption tomogram was measured with a diode for mineral density distribution assessment. The high resolution data acquired at the beamline allows us to gain precise structural knowledge of the sample, and evaluate the contribution of nanoscale tissue properties to the strength of human bone at the microscale.

Sample preparation and previous testing

A total of 5 cadeveric femoral necks of various ages (57-93) were used in the preparation of this experiment. From each femoral neck, two micropillar samples (80-100um diameter) were extracted from the osteonal bone on the superior and inferior sides respectively. The samples were measured prior to the X-ray experiment by microcompression using a cyclic loading and unloading protocol for characterization of the elastic modulus and the yield point. Additionally, the pillars were measured by Raman Spectroscopy to extract information on the chemical structure. As expected, the mineral to matrix ratio (ratio of the integral areas of v2PO4/amide III) of the bone dominates the mechanical properties. This quantity was positively correlated with the measured elastic moduli, yield strains and stresses. The structure of the sample measured by SWASTT in the current experiment is currently being analysed in the aim of identifying structural differences accounting for the remaining variability in the mechanical properties of the samples.

Samples measured and quality of measurement/data

During the beamtime, 4 micropillars (2 pairs of samples, ages 57 and 89) were succesfully measured. The micropillars were raster scanned with an x-ray energy of 13.2keV, a beam and step size of 2um and exposure time of 5ms. The scattering data was recorded at a detector distance of 200mm, leading to the measurements of scattering vectors in the full azimuthal range between 0.2 – 30nm-1. This setup allows for simultaneous measurement of the orientation of the SAXS and WAXS signals, arising from the nanoscale mineral platelets and the mineral crystals respectively. In figure 1, the bone scattering is shown with the principal SAXS (orange) and WAXS (blue) orientations. Combined with an additional absorption tomogram measurement, the samples were fully characterized by high-resolution SWASTT and absorption tomography as planned. The measurement time per sample was ~10h, which was longer than initially expected, due to the extra time needed for the separate absorption measurement and the use of an increased field of view. The latter was necessary due to the final sample geometry and the sub-optimal recentering of the sample after each projection. In addition we lost some time due to detector software problems and establishing means of acquiring absorption data, crucial for the reconstruction. Thus only 2 pairs of samples were measured from the prepared 5 pairs.

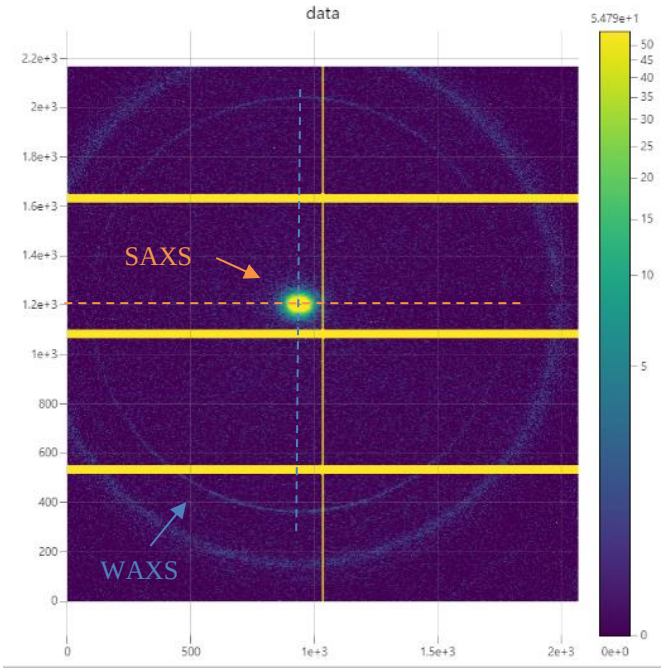

Figure 1 : Bone scattering pattern, illustrating the simultaneous SAXS and WAXS measurement.

Plan and status of data evaluation

All the collected data is currently being processed. The scattering datasets were radially integrated into 8 azimuthal and 1024 radial bins (figure 2) using a custom made python script. In figure 2 (right), the scattering intensity in two orthogonal directions is plotted, displaying the anisotropy at both length scales. These orientations correspond to those indicated in current bone models [1]. SWASTT reconstructions will be made in specific q-bins, corresponding to the SAXS and WAXS orientations, simirlarly to the evaluation of data previously measured at the beamline [2]. In a second step, we will reconstruct fully q-resolved reconstructions which provide the orientations across all probed length scales. The structural information at each length scale will be correlated to the mechanical properties and compared between sample, in the aim of identifying key structures influening the bone strength. Currently, the absorption tomograms are under reconstruction. They will serve as starting point for the alignment of the scattering data in the reconstructions.

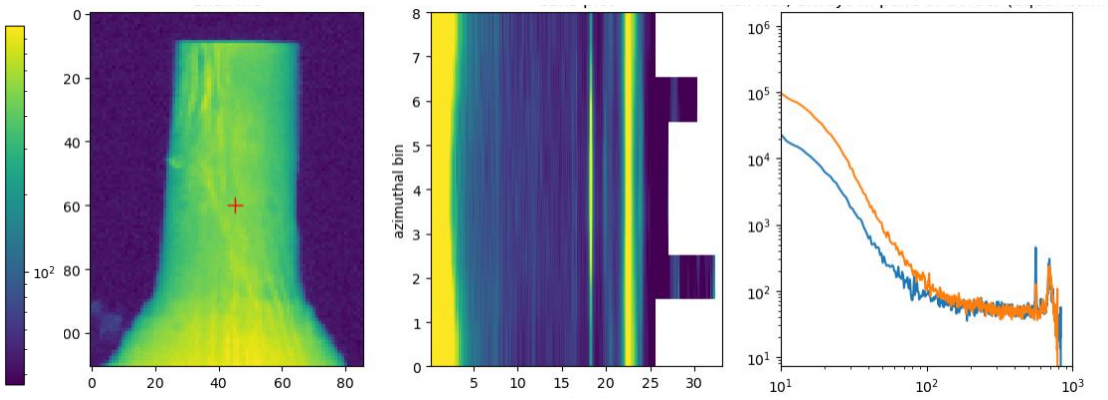

Figure 2 : Left: Symmetric Intensity of scattering signal, illustrating structural variation within the sample. Middle: Cake plot displaying the radially integrated intensity in 8 azimuthal segments. Right: Intensity of two orthogonal segments, representing the principal SAXS (orange, horizontal) and WAXS (blue, vertical) orientations.

[1]: Reznikov N, Shahar R, Weiner S., Acta Biomater.10(9):3815-26 (2014)
[2]: Grünewald, T. A., Liebi, M., Wittig, et al., Science Advances, 6(24), 4171–4183 (2020)