EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: 3D mapping of hydroxyapatite orientation at the bone-cartilage interface by X-ray Diffraction Computed Tomography (XRD-CT)	**Experiment number**: LS-2810
Beamline: ID15A	**Date of experiment**: from: 27 October 2018 to: 30 October 2018	**Date of report**: 10/09/2020
Shifts: 9	**Local contact(s)**: Marco Di Michiel	*Received at ESRF:*

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Report:

X-ray diffraction tensor tomography (XRDTT) experiments were performed according to the plan in the beamtime proposal. Two samples consisting of porcine bone and cartilage of approximate dimensions 2 x 2 x 2 mm^3 were measured with XRDTT. Additionally a third bone and cartilage sample and a sample of Pierre shale 1 were measured with X-ray diffraction computed tomography (XRD-CT), using similar setup as for XRDTT using a single tomography axis. Complementary propagation phase-contrast CT (PPC-CT) and attenuation contrast CT was performed after the XRDTT/XRD-CT experiments.

For XRDTT a multi-axis rotation stage was used to tilt the sample around a horizontal axis while rotation of the sample around a conventional tomography axis. A total of 65 × 67 points in (x,y) were collected per projection, with a step size of 50 µm in x and y. For each sample, 259 projections were obtained from combination of projection angles $\beta \in [0, 40°]$ and $\alpha \in [0, 360°]$. For $\beta = 0$, equivalent to ordinary perpendicular-axis tomography, 61 projections with $\alpha \in [0, 180°]$ were obtained. For $\beta \in [5, 40°]$ a reduced number of projections were measured compared to $\beta = 0$. A total number of 65 × 67 × 259 = 1,127,945 diffraction patterns were collected. An exposure time of 50 ms for each diffraction pattern was used, giving a total exposure time of 15.2 hours. With additional overhead time for motor movements, the total measurement time was approximately 26 hours.

For the shale sample XRD-CT measurements were done by using a single tomography axis, i.e. $\alpha = 0$, cf. Figure 1a. The beam was line scanned in x while the sample was rotated about y between each line scan. The scanning direction was reversed between each line scan, to reduce motor movement time. 71 steps in x and a total number of 61 angular steps of $\beta \in [0, 180°]$ were used ($\Delta\beta = 3.0°$). The scan procedure was repeated for 29 steps in y to measure the full 3D volume, effectively giving 74 × 61 × 29 = 155,559 recorded diffraction patterns for the full sample. An exposure time of 30 ms was used. The total measurement time was approximately 2 hours, including over-head time for motor movements.

Hydroxyapatite (HA) crystallites in the bone regions of the samples provided oriented diffraction signal which could be used for tensor tomography reconstruction of the local orientation of the HA crystallite *c* axis orientation within the bone/cartilage samples, illustrated in Figure 1. For the shale sample the XRD-CT provided spatatially resolved chemical information about the sample and in addition the clay mineral orientation could be studied, as shown in Figure 2.

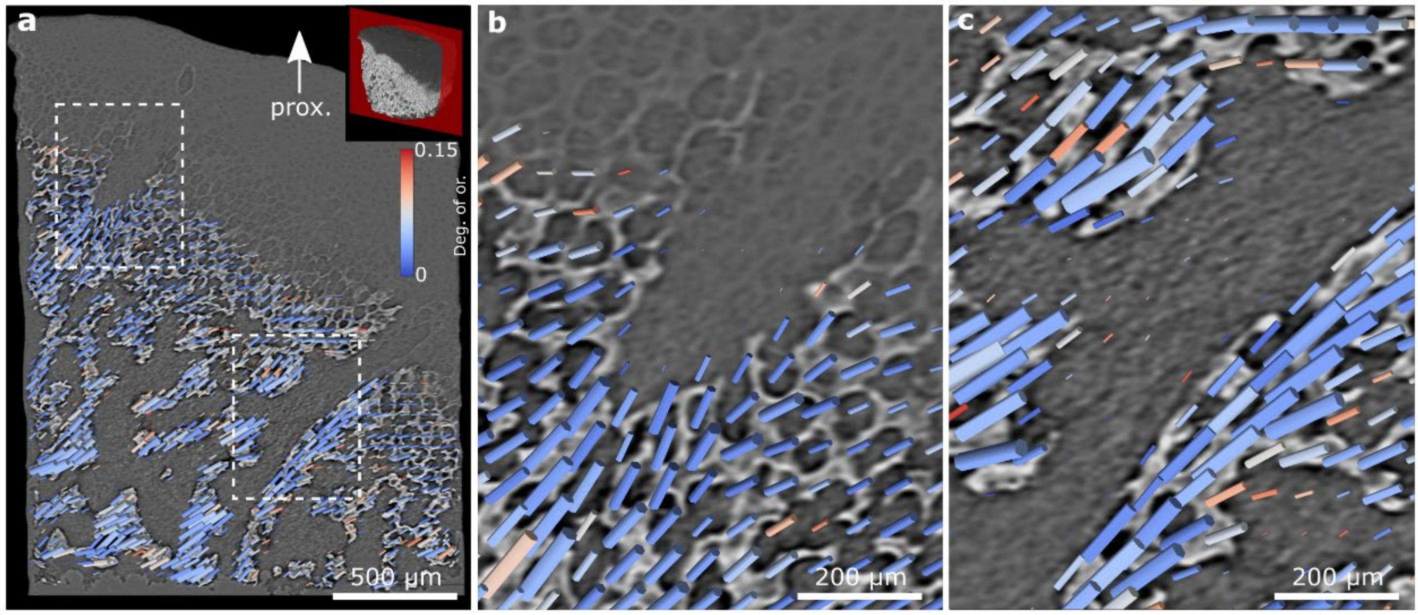

Figure 1: Hydroxyapatite crystallite orientation in a bone and cartilage sample from a pig, obtained by XRDTT. a) Cross-section of the sample. b-c) Magnified sections corresponding to the dashed boxes in a). The colored cylinders indicate the preferred orientation of the hydroxyapatite c axis, where the cylinder colors correspond to the calculated Hermans' orientation parameter S. The orientation maps have been registered with ppc-CT tomograms. X-ray diffraction tensor tomography and ppc-CT was performed at ID15A. Figure adapted from Mürer et al. Abbreviations: cc: cartilage canal. prox.: proximal.

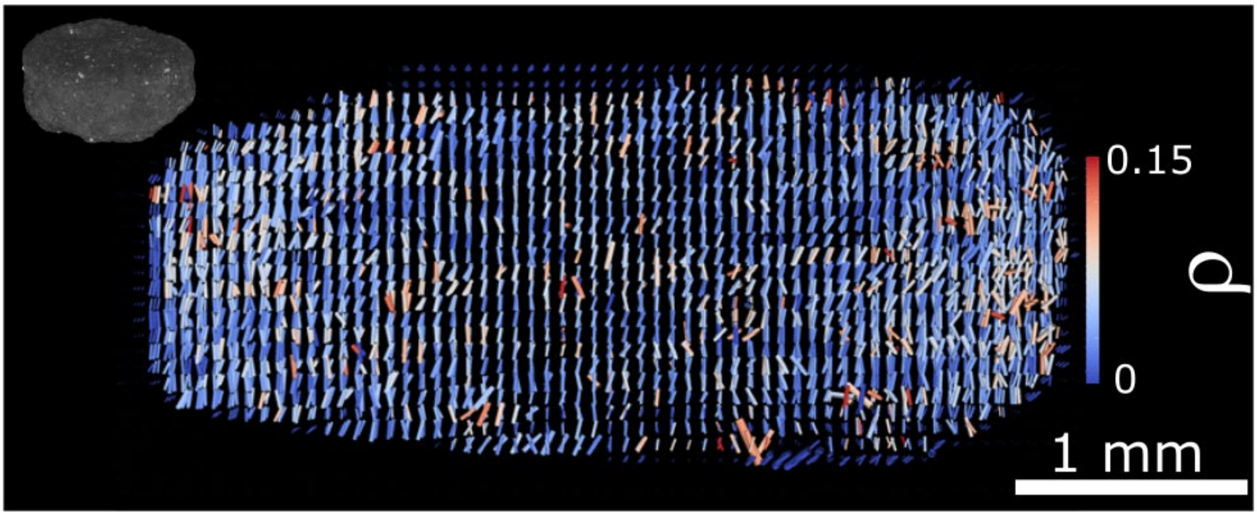

Figure 2: Orientational imaging of clay mineral orientation in a sample from Pierre shale 1. a) Attenuation contrast CT reconstructed volume of sample measured. b) Orientation of chlorite clay mineral crystallites in sample cross section corresponding to the indicated plane in a). The color coding corresponds to the reconstructed degree of orientation ρ.

The suggested experiments have resulted in the following scientific manuscript to be published:

Mürer, F. K. et al, *"Quantifying the hydroxyapatite orientation near the ossification front in a piglet femoral condyle using X-ray diffraction tensor tomography",* Submitted.

Mürer, F.K. et al. "3D checmical and orientational imaging of Pierre shale using X-ray diffraction computed tomography", *In preparation.*

Beamline configuration:

XRD-CT / tensor XRD-CT:

Beam height: 50 µm
Beam width: 50 µm
Energy: 50.00 keV
Wavelength: 0.2480 Å
Detector: Dectris Pilatus3 X CdTe 2M 20-bit. 1475 x 1679 pixels (horizontal × vertical), with a square pixel size of 172 µm.
Sample-detector distance: 800 mm

The detector was placed to capture a momentum transfer q-range of 0.20 $Å^{-1}$ to 6.0 $Å^{-1}$ and as to obtain as much as possible of the hydroxyapatite 002 peak (q = 1.82 $Å^{-1}$) with little overlap with the detector module gaps. The pencil-beam was collimated to a cross section of dimensions 50 × 50 $µm^2$ using compound refractive lenses and slits. The detection area consisted of 1475 x 1679 pixels (horizontal × vertical), with a square pixel size of 172 µm. A CeO_2 powder sample was used for calibration.

PPC-CT / attenuation contrast CT:

Beam height: 3.50 mm
Beam width: 7.64 mm
Energy: 50.00 keV
Wavelength: 0.2480 Å
Detector: 2401 x 1101 pixels (horizontal × vertical), pixel size: 3.18 µm
Sample-detector distance (PPC-CT /att. CT): 2300 mm / 300 mm.