ESRF	**Experiment title:** Stopping caries before it begins: imaging 3D tooth texture in (remineralised) sub-surface lesions by energy dispersive Laue diffraction	**Experiment number**: 32-02-803
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Report:

In this experiment we aimed at unveiling the impact of beginning tooth decay (sub-surface lesions) and remineralisation treatment on 3D crystallographic texture in human dental enamel. For this purpose we used the novel and recently established method of energy dispersive Laue diffraction (EDLD) texture scanning [1] on tooth samples affected by artificially induced caries-like subsurface lesions, remineralised lesions, and healthy tooth specimens with micrometer resolution.

For this purpose we brought a pixelated energy dispersive Camera (SLCam, ov type pnCCD, owned by University of Ghent) and mounted it in the beampath on to a custom made rack including a constructin that allowed tilting and rotating of the detector. This was necessary to be able to cover a range of 2theta of up to

45° with the very small chip available (approx. 1x1 cm) in order to take advantage of the quasi 3D information provided by the use of a white beam and energy dispersive approach in EDLD.

Samples were mounted in very closely in front of the beam stop. The sample-detector distance was approx. 1.5 cm and we used a focused pink beam with a spot size 2 µm.

Several samples were investigated. All of them contained enamel in one of the following conditions: 1) demineralized artificial sub-surface lesion; 2) artificially remineralized; 3) healthy enamel. Samples had been ground to thin sections before the experiment and mounted between two silicon nitride windows (Fig. 2a) and measured in transmission.

Since the online microscope only allowed to view the sample at an angle of approx 45°, we used a fluorescence detector to map the exact sample location in the beam by recording the Ca fluorescence line indicating mineralized tooth material. Subsequently, line tracks were scanned across the dentin-enamel interface for all samples.

In summary, 4 samples were scanned with 2 line tracks, each extending from the enamel into the dentine.

Fig. 1: *Experimental setup including the SLCam, own custom made beam stop and rack to hold the SLcam and allow movement of the camera to cover 4 quadrants.*

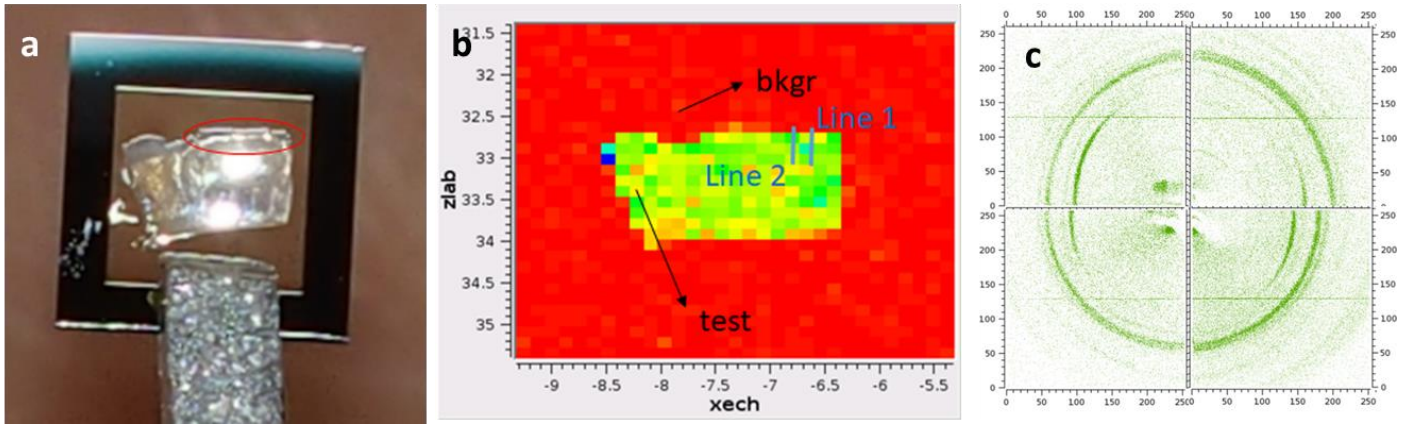

Fig. 2: *EDLD on healthy tooth enamel. (a) silicon nitride window holding a thinly ground section of a tooth. The area marked with a red oval is the region of interest with the enamel and dentin enamel junction (DEJ). (b) prior to measurement a Ca fluo map was recorded. The blue lines indicate the position of linear scans performed across the enamel and DEJ. (c) tpyical diffraction pattern showing the peaks of hydroxyapatite (HAP) and reveiling strong texture. Note that the pattern consists of 4 quadrants, corresponding to 4 different tilt/rotation positions of the SLCam on the rack to cover as much of reciprocal space as possible. The offset is then internally corrected by an evaluation algorithm.*

Data are currently being evaluated. Data treatment took some time due to the need to develop specific algorithms to deal with the data cubes (2D scattering images with every pixel containing an energy spectrum) and extract texture information. Results will be published in a scientific journal.

References:

[1] Grünewald, T.A. et al., Angew. Chemie IE. 2016, 55(40) 12190–12194.