•••••	Experiment title:	Experiment
	2D Mapping of Dental Enamel Affected by Caries	number:
ESRF		28 19 11
Beamline:	Date of experiment:	Date of report:
BM28	From: 15 Dec 2010 to: 17 Dec 2010	26/02/2011
Shifts:	Local contact(s):	Received at ESRF:
6	Miss Gemma Newby	
Names and affiliations of applicants (* indicates experimentalists):		
Al-Jawad*. M. Anderson*. P. Siddiqui*. S. Department of Dental Physical Sciences, Queen		
Mary University of London.		

## **Report:**

Dental enamel is the most highly mineralised and strongest biological hard tissue. It comprises 95% hydroxyapatite (HA) mineral, 5% water, and 1% organic matter (non-collegenous protein). The hydroxyapatite crystal structure of dental enamel has been determined previously by several workers. HA has space group P6<sub>3</sub>/m with lattice parameters a=9.513Å and c=6.943Å.

Dental caries is a worldwide problem affecting 60-90% of school children and many adults, being part of the fourth most expensive disease around the world, it is absolutely essential to get a greater understanding of the processes in order to aid caries prevention. The objective of our recent experiment conducted on XMaS was to observe the changes that occur in texture and preferred orientation in carious enamel, more imporatantly identifying the effects of remineralisation on texture.

We used the MAR CCD area detector and the travelling sample platform to collect diffraction images every 20-150 $\mu$ m from various sections of teeth ~500 $\mu$ m in thickness. The six shifts were used to collect diffraction images from enamel with demineralised artificial lesion (both 1 and 2 weeks), artificially remineralised enamel and naturally lesionous enamel tissue. We looked at different regions on each sample, including an internal control "healthy" region in each affected tooth section.



## Figure 1. Intensity versus Azimuthal

Example of azimuthal peaks taken from the 002 reflection of a diffraction patterns of the DM3 sample. The data points show the azimuthal angle curve for the 002 reflection, the pronounced peaks highlight a high degree of texture in this sample. Both peaks have been fitted to a Gaussian plus baseline (red and green lines respectively).

Different region of the treated tooth samples were scanned on the XMaS beamline. The 2D diffraction patterns were analysed using Fit2D. For a first approximation before further in depth analysis, texture direction in the 002 reflection can be determined by measuring the change in full-width half maximum of the texture peaks around the Debye ring of that reflection. Figure 1 is an example of the Intensity versus Azimuthal angle around the 360° Debye ring of the 002 reflection. The pronounced peaks represent the 002 arcs. By fitting these peaks to a Gaussian peak shape the orientation of the crystallite can be determined by extracting the full width half maximum (FWHM) values. Figure 2 shows a plot of these FWHM values as a function of distance from the enamel surface for three samples: A healthy unaffected control (Control); a tooth section demineralised for one week (DM3); and a tooth section demineralised for one week and then left in remineralising solution containing fluoride for one week (RMF5).



## Figure 2. Changes in the FWHM.

One track taken through the tooth section going from enamel surface to EDJ from each sample (demineralisation, remineralisation and control) showing the change in full width half maximum of the first peak.

A systematic decrease in DM3 with increasing distance from the enamel surface is an indication of a rise in texure (or preferred orientaion) of the hydroxyapatite crystallites until about 275µm into the enamel (Fig 2). This is an indication that there is less texture (where the carious lesion occurs), the decreasing width of the Braggs peak up to 250µm shows greater order of texture travelling away from the surface. The similarity between thte two RMF5 and control plots is a point of interest as it may indicate that remineralised enamel can mimic healthy enamel, however further analysis is required to reinforce this. There is a significant difference between the FWHM value between DM3 and RMF5, the latter being considerably lower showing that there is a higher degree of texture around the surface of the enamel. Additionally there is evidence of uniformatiy in the RMF5 section as the the width remain more or less constant. The results thus far seem to be promising as they follow the expected trends and agree with previous work carried out on enamel affected by caries [1].

Further analysis is currently in progress, we are looking at more tracks along each sample before using Rietveld refinement for each diffraction pattern to determine the calcium phosphate phases and closely observe changes in texture and lattice parameters

[1] Yagi. N. Ohta. N. Tanaka. T. Terada. Y. Kamasaka. H. To-o. K. Kometani. T. Kuriki. T. 2009. Evaluation of Enamel Crystallites in Subsurface Lesion by Microbeam X-ray Diffraction. *Journal of Synchrotron Radiation.* 16 398-404