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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** 3D Crystal Orientation in Human Dental Enamel	**Experiment number**: 28-01-832
Beamline:	**Date of experiment**: from: 03 Sept 2008 to: 09 Sept 2008	**Date of report**: 30 March 2009
Shifts: 18	**Local contact(s)**: Laurence BOUCHENOIRE	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The hydroxyapatite (HA) structure of human dental enamel has been determined using powder x-ray diffraction, and is well established as space group P63/m with lattice parameters a=9.441(2)Å and c=6.878(1)Å. However, these values were obtained from measurements of powdered enamel collected from several teeth, and as a result any information on the spatial variation of the texture relating to the growth of the HA crystallites was lost. The HA crystallites are laid down by ameloblasts as nanorods which are grouped into cluster called prisms. Prisms are approximately 5µm in diameter and up to millimetres long and are arranged with their long axes at acute angles to the enamel-dentine junction (EDJ). After the maturation stage of enamel formation the ameloblast cells are broken down which means that, unlike bone, dental enamel in not able to regenerate itself. The orientation of prisms in enamel has been studied in the past using electron microscopy. Although this is a valuable tool for finding the prism shape and size in a particular plane of enamel, it is a qualitative technique and does not give quantitative information on the degree of alignment in different parts of a tooth in any detail. Understanding the spatial texture distribution and orientation of HA crystallites in the dental enamel of a tooth is extremely valuable in the clinical setting where this knowledge would improve cavity drilling techniques (for example to avoid weakening of the tooth by drilling at a particular angle which leaves the ordered enamel crystallite arrangement intact). In addition this crystallographic information ultimately will need to be more fully understood if functional, synthetic, biomimetic enamel is to be developed.

For this XMaS experiment we were interested in characterising the texture in dental enamel in 2D and 3D from several different tooth-types to give us a complete picture of texture in the permanent dentition. In addition we compared fully mature enamel from a healthy molar tooth, to a specimen of immature enamel taken from an archaeological sample of a young human in order to determine whether textural analysis can provide useful insights into the maturation stage of dental enamel formation. The preliminary analysis of these data is given below.

Diffraction patterns were taken at intervals of 150 µm resolution for the immature enamel (Figure 1a) and 300 µm for the mature enamel (Figure 1b), for 500 µm thick tooth sections. The texture directions for the immature enamel and mature enamel tooth sections are shown in Figure 1 a) and b) respectively. It can be seen from this that for both immature and mature enamel the texture direction in the 002 reflection is approximately perpendicular to, and follows the contour of the enamel-dentine junction. From our knowledge of the structure of dental enamel it would appear that the preferred orientation in the 002 direction approximately follows the direction of the enamel prism arrangement.

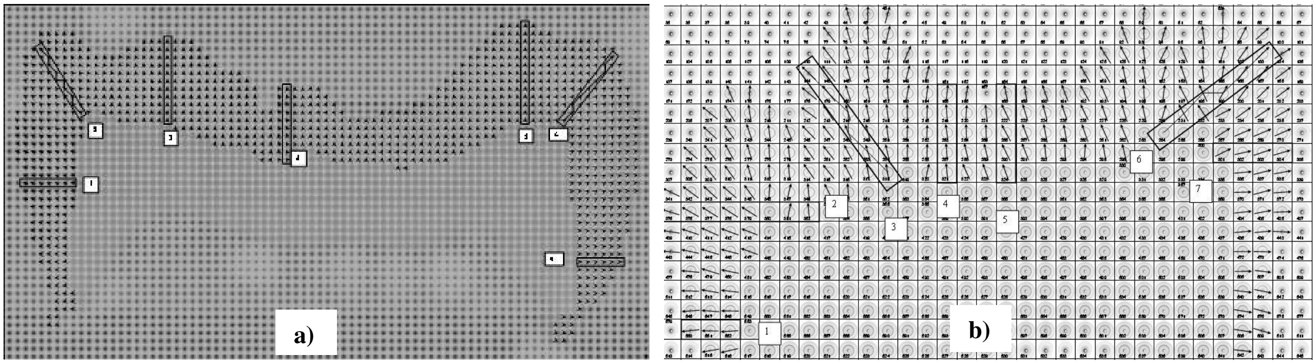

Figure 1 – Texture direction of the 002 reflection of HA in a) immature enamel and b) mature enamel

For particular tracks through the enamel, the FWHM of the intensity pattern around the Debye ring of the 002 reflection was used to evaluate the the degree of crystallite alignment – the higher the FWHM value, the less ordered the crystallites and vice versa. Taking each 2D diffraction image, the intensity was integrated over 360º in a narrow band containing the 002 reflection and plotted versus the azimuthal angle. The value for the FWHM was obtained by fitting the two peaks to a Gaussian peak shape. The results of this analysis have been plotted in Figure 2 a) and b).

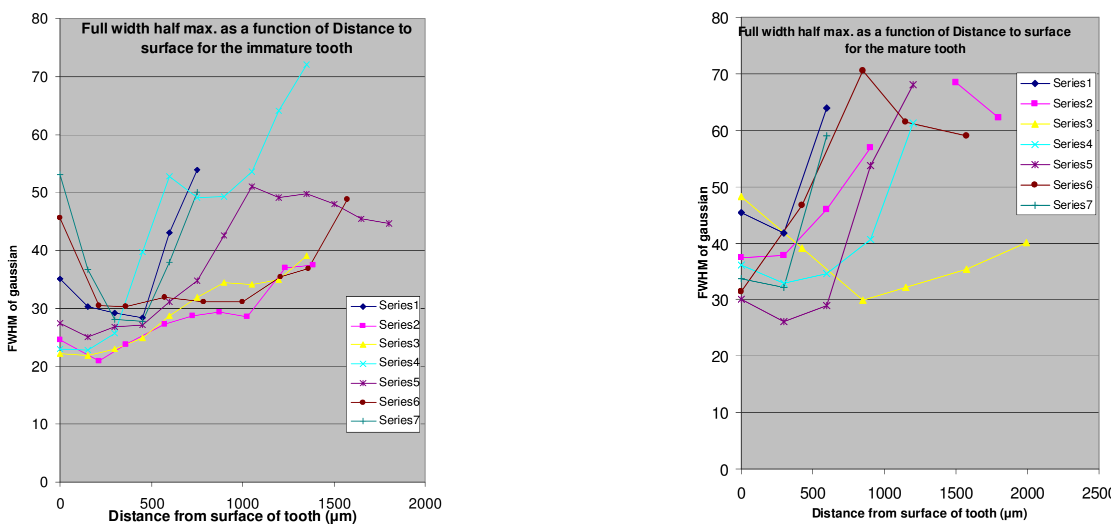

Figure 2 – Several tracks through sections of a) immature enamel and b) mature enamel going from enamel surface to EDJ showing the change in FWHM of the 002 reflection azimuthal angle curve as a function of distance from the enamel surface. The tracks are indicated in Figure 1. The solid lines are a guide to the eye.

The general trends seen in Figure 2 suggest that for both immature and mature enamel there is a higher order of crystallite alignment in a region ~300µm from the surface enamel, and that as you track deeper into the enamel towards the EDJ the crystallites become less ordered. Our preliminary results suggest that the texture distribution varies more in mature enamel than in immature enamel – possibly indicating a larger distribution of crystal sizes in the fully formed enamel as compared to the immature enamel. The results also suggest that on average the HA crystallites in the immature enamel are more aligned as compared to fully mature enamel. This may be due their particular stage of maturation where the needle-like crystallites have formed to their full length but not yet filled out in their width. Further interrogation of the data and further experimentation will be necesaary to confirm these findings.