EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

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 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:** Optimisation Of Restorative Materials Via 3-D Strain/Texture Analysis Of Dental Enamel	**Experiment number**: 28 01 732
Beamline: BM28	**Date of experiment**: from: 02 Sept 2005 to: 06 Sept 2005	**Date of report**: 26/02/2006
Shifts: 12	**Local contact(s)**: Laurence Bouchenoire	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

M. Al-Jawad* and R. Cywinski*, School of Physics and Astronomy, University of Leeds, Leeds; S.H. Kilcoyne*, Institute for Materials Research, University of Salford, Salford; D. Wood* and R.C. Shore*, Leeds Dental Institute, Leeds

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 $P6_3/m$ with lattice parameters a=9.513Å and c=6.943Å. In all previous reports, the measurements were made on powdered enamel collected from many teeth, therefore any texture information regarding the growth of the HA crystallites was lost. This information however is extremely valuable in the understanding of the formation of enamel, and in improved design of dental composites for restorations. Our recent experiment on XMaS for the first time maps the change in preferred orientation as a function of position in an intact section of tooth. We used the MAR ccd area detector and the travelling sample platform to collect diffraction images every 50-300μm from various sections of teeth ~500μm in thickness. Over the four days we collected diffraction images from healthy teeth, lesionous teeth, teeth from high fluoride water areas and teeth with genetic defects (amelogenesis imperfecta).

An example of a 2-D map is shown in Figure 1.

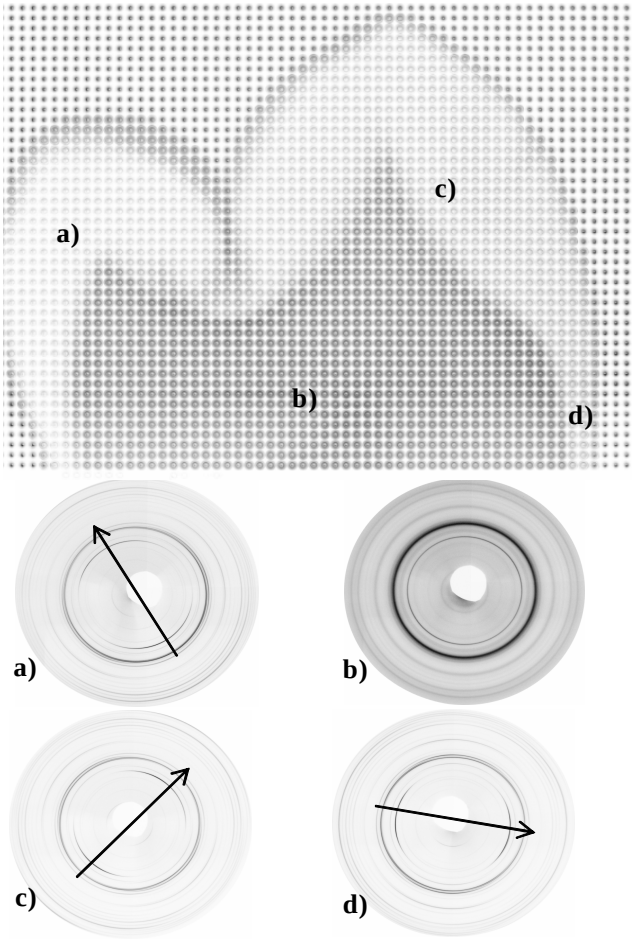

Figure 1. 2D image of whole tooth section of a healthy second molar. Diffraction images were collected every 150µm. a), c) and d) illustrate the change in texture direction at difference positions within the enamel. b) shows the poorly crystalline nature of dentine.

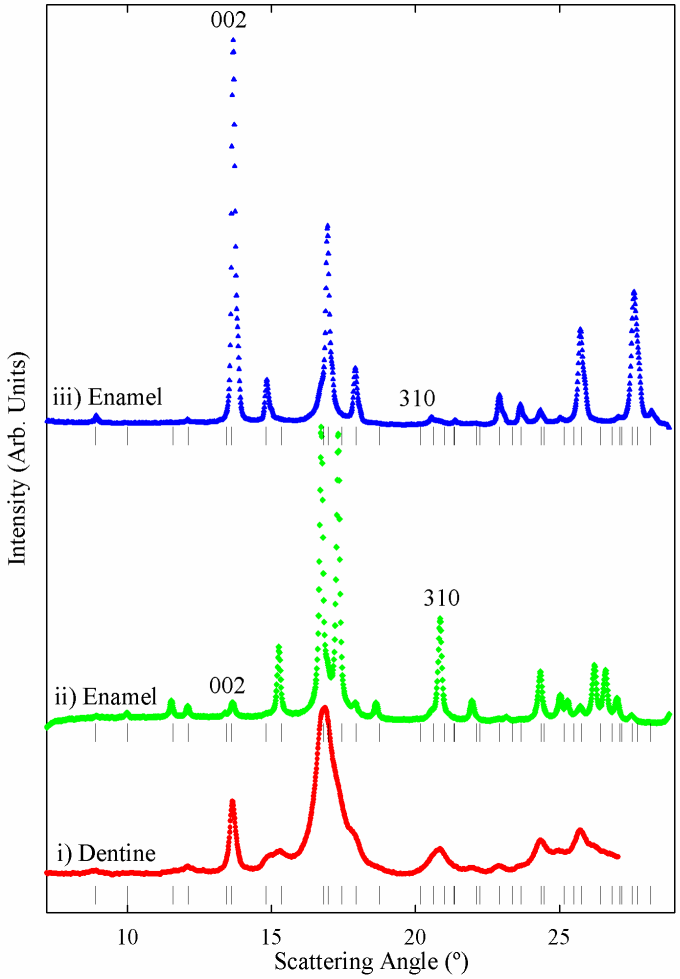

Figure 2. Diffraction patterns generated from 2D images. Each pattern is a 20º cake slice of the full 360º images. i) poorly crystalline dentine (at position b) in Fig 1), from 2θ=-10º to 10º ii) enamel at position d) in Fig 1) from 2θ=-10º to 10º and iii) enamel at position d) in Fig 1) from 2θ=80º to 100º.

The regions of highly crystalline enamel and poorly crystalline dentine are clearly visible. Additionally, it can be seen that below the groove between the two tooth cusps (the fissure), there is a circular region of enamel which is less crystalline than the surrounding structure. We believe this to be caused by a fissure lesion in the enamel which has affected the crystalline structure of the enamel. This type of lesion is undetectable by inspection of a whole tooth that has not been sectioned.

Diffraction patterns generated using fit2D from images b) and d) in Figure 1 are shown in Figure 2. Two orthogonal peaks 002 and 310 are marked in order to compare their peak intensities. In polycrystalline HA the value of the ratio of 002/310 should be between 2-3. In this highly textured enamel, the value of 002/310 varies enormously between 0.14(3) for Fig 2 ii) and 92.6(1) for Fig 2 iii). We are currently using Rietveld refinement of each diffraction pattern with texture and strain parameters included to produce complete 2-D texture and strain contour maps of tooth enamel. Preliminary results suggest that the strongest preferred orientation was found along the 002 direction. Cuspal areas of high crystallite alignment match the expected occlusal surfaces of the teeth. Within individual teeth different cusps had quite different intrinsic strain profiles, which would agree with previously published theoretical calculations where independent cuspal movement was described. Such results reveal new information on the texture and strain profiles within teeth which may have potential in the future to optimise dental composite materials for restorations.