Three Dimensional Mapping of Texture in Dental Enamel

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Abstract. We have used synchrotron x-ray diffraction to study the crystal orientation in human dental enamel as a function of position within intact tooth sections. Keeping tooth sections intact has allowed us to construct 2D and 3D spatial distribution maps of the magnitude and orientation of texture in dental enamel. We have found that the enamel crystallites are most highly aligned at the expected occlusal points for a maxillary first premolar, and that the texture direction varies spatially in a three dimensional curling arrangement. Our results provide a model for texture in enamel which can aid researchers in developing dental composite materials for fillings and crowns with optimal characteristics for longevity, and will guide clinicians to the best method for drilling into enamel, in order to minimize weakening of remaining tooth structure, during dental restoration procedures.

Introduction

The hydroxyapatite structure of human dental enamel has been determined using powder x-ray diffraction, and is well established as space group P63/m and lattice parameters a=9.441(2)Å and c=6.878(1)Å [1–3]. 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. Hirota examined the tilting of the enamel-prism orientation in a human canine using laboratory two-dimensional x-ray diffraction [4]. However, only 12 points within the tooth were measured and therefore the information obtained about the prism orientation does not relate to the whole tooth. Understanding the spatial texture distribution and orientation arrangement in the dental enamel of a tooth is extremely valuable in order to improve dental restorative materials and techniques and ultimately will need to be more fully understood if functional, synthetic, biomimetic enamel is to be developed. Recently, we have shown for the first time how synchrotron x-ray diffraction can be used to determine the basic crystallographic parameters of the HA phase across a whole intact tooth section, allowing us to explore composition and texture on the sub-millimetre length-scale. We found that the orientation of the enamel crystallites in a bucco-lingual section of a lower second premolar (LR5) followed the shape of the enamel-dentine-junction (EDJ) with their long axes approximately at right-angles to the EDJ, and that areas of high crystallite alignment on the tooth cusps matched the expected biting surfaces [5]. We now present detailed results of the texture direction and magnitude in an intact mesio-distal section, and we show the change in texture direction in three dimensions by studying several sequential mesio-distal sections through the same tooth.

Experimental Procedure

The tooth sections used in this study were from a maxillary first premolar (UL4). The tooth was collected with informed consent from a patient undergoing routine orthodontic extraction at the Leeds Dental Institute. The extracted tooth was sterilised by autoclaving prior to storage at 4 ºC in a thymol-saline solution to prevent bacterial growth. It was cut into six 500 µm-thick longitudinal sections perpendicular to the mesial and distal surfaces. Synchrotron x-ray diffraction measurements were taken on XMaS [6] at the European Synchrotron Radiation Facility (ESRF) using an x-ray wavelength of λ=0.82 Å (equivalent to an x-ray energy of 15 keV). Vacuum tube slits were used to focus the x-ray beam to a diameter of 300 µm on the sample. The tooth sections were mounted in transmission geometry onto a traveling sample platform such that they could be scanned in two orthogonal directions perpendicular to the beam. A 2D detector with 2048x2048 pixel resolution was mounted behind the sample and perpendicular to the incident beam for the collection of 2D diffraction images. Diffraction images were collected every 5 s therefore a 300 µm-resolution map of the tooth could be collected in approximately 2 hours. Texture contour maps were produced by performing Rietveld refinement analysis, using an inhouse batch processing program and GSAS software, and extracting intensity (texture) coefficients [7].

Results and Discussion

A composite map of diffraction images from the central mesio-distal tooth section is shown in Fig. 1. The darker patterns in the middle of the figure are from dentine and the lighter patterns covering these are from the enamel. An example of an individual diffraction image from the enamel region is shown in Fig. 2. The intensity pattern around the Debye ring of the 002 reflection was used to evaluate the texture direction, and this is shown as the solid lines in Fig. 1 and Fig. 2.

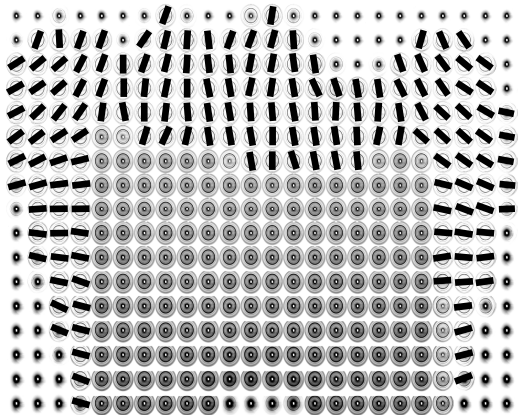

Fig. 1 Composite image of central mesio-distal slice with lines showing the 002 texture direction

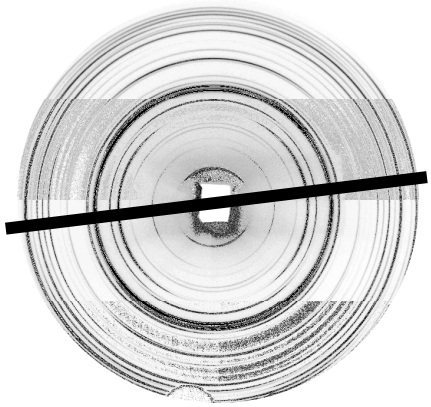

Fig. 2. Individual 2D diffraction image illustrating the 002 texture direction

In contrast to the bucco-lingual slice evaluated previously [5], it appears that in this tooth section the preferred orientation in the 002 direction does not consistently follow the contour of the EDJ. Instead it is perpendicular to the EDJ along the sides of the tooth in the cervical enamel and in the central part of the tooth crown, while on either side of the tooth cusps the 002 texture direction is parallel to the EDJ. This surprising since it implies that the 002 texture direction is not the same as the prismatic growth direction in this mesio-distal tooth section. The 002 texture direction was evaluated for the remaining five mesio-distal sections and the angle of the 002 texture direction has is plotted as a function of the distance from the buccal end in Fig. 3. The main plot in Fig 3 shows variation in texture direction in nine tracks on the mesial side of the tooth. The inset to Fig. 3 is a 3D model of the tooth showing the change in texture direction in three dimensions in the 002 reflection. Each shaded strip represents one of the six tooth sections measured. The data suggest a curling of crystallite orientation both along the length of the section, and also through a series of

sections. This 3D curling of the texture through the tooth may explain the unusual texture direction seen on the cusps of the central mesio-distal section presented in Fig. 1.

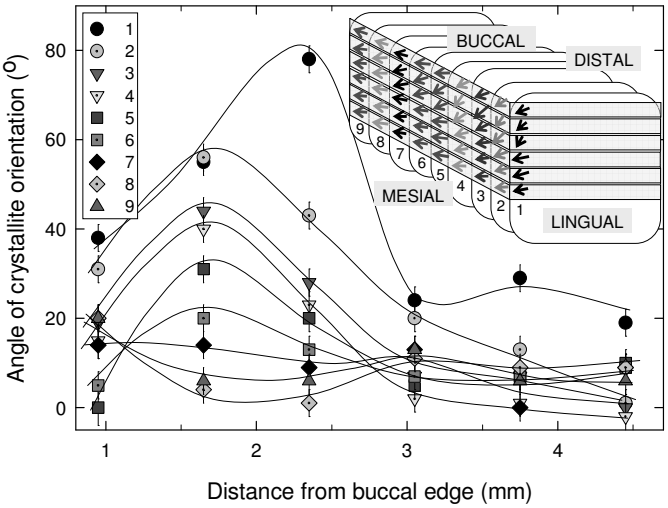

Fig. 3 Crystallite orientation as a function of distance from the buccal end. The inset shows a 3D model reconstruction of the texture direction through the whole tooth.

The magnitude of preferred orientation in the central mesio-distal tooth section has been quantified using Rietveld refinement. Each 2D diffraction image was sliced into 5° slices and the vertical slices used to evaluate the texture in the 002 reflection relative to the vertical. An example of a typical 1D Intensity vs 2θ diffraction pattern together with the calculated pattern is shown in Fig. 4.

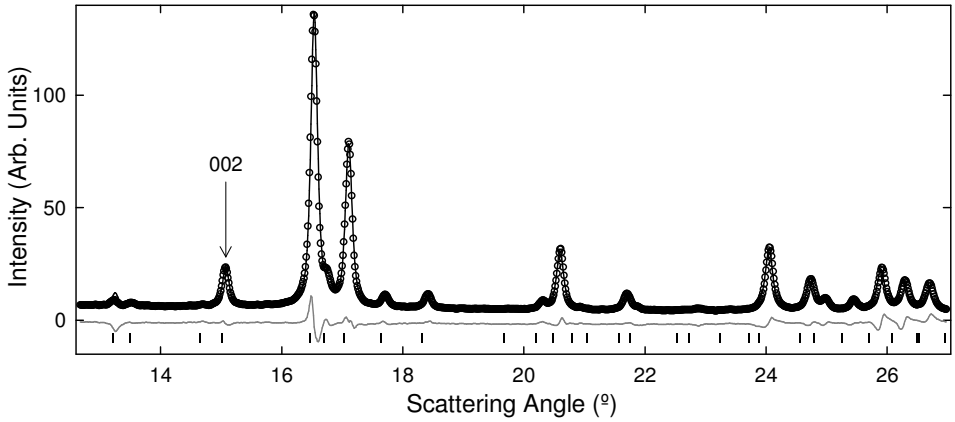

Fig. 4 Typical Rietveld refinement of 1D diffraction pattern taken from a mesio-distal section.

The open circles are the observed data points, and the solid line is the calculated diffraction pattern. Below the pattern is a plot of the difference (observed–calculated), and beneath are the tick marks for the calculated diffraction pattern of HA. The difference plot shows that the agreement between observed and calculated data is generally very good with a typical value for χ^2 of 1.5. Similar refinements were carried out on all diffraction patterns, and the 002 preferred orientation parameters extracted from the intensity of the 002 reflection (labelled in Fig. 4). A contour map showing the change in magnitude of preferred orientation in the 002 reflection is plotted in Fig. 5. Areas with higher values of preferred orientation parameter are more strongly textured i.e. the crystallites are more aligned to the vertical direction in these areas. Areas with a low texture coefficient have crystallites less well aligned to the vertical. It can be seen in Fig. 5 the HA crystallites are most aligned at the top of the tooth crown. Conversely, along the sides of the tooth away from the cusps generally the crystallites are not well ordered in the vertical direction – which is what we would expect from the texture direction map in Fig. 1 which shows the crystallites aligned to the horizontal at the sides of the tooth crown. In normal centric occlusion, the maxillary first premolar will be occluded by both the mandibular first and second premolars [8]. It is interesting to observe that the areas of high crystallite alignment at the top of the crown match the expected occlusal surfaces.

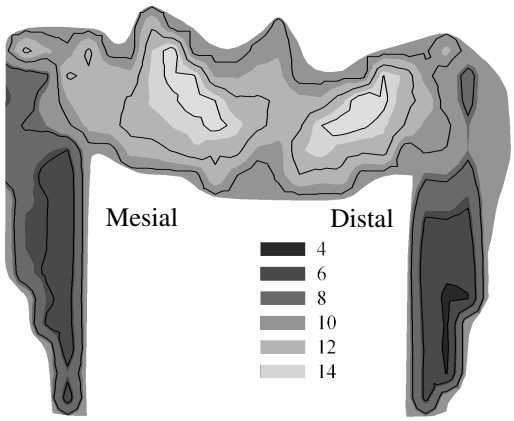

Fig. 5 Texture distribution map generated from the calculated texture coefficient via refinement.

Conclusions

We have studied the direction and magnitude of texture as a function of position within several intact tooth sections of a maxillary premolar. From our 3D model reconstruction, our results suggest there is a 3D curling of the crystallite orientation in both the mesio-distal and bucco-lingual directions. We have shown through this work that synchrotron X-ray diffraction is a powerful technique in the study of the crystallography and microstructure of dental enamel and will allow us to completely characterize the crystallographic properties of dental enamel in 3D and therefore allow optimized design of dental restorative materials and procedures. This technique could be equally successful in the study of other biological hard tissues, bioceramics, and biological-synthetic complexes.

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