Nondestructive 3D-evaluation of human dentin by microtomography using synchrotron radiation

Z V Gudkina\textsuperscript{1,2}, T S Argunova\textsuperscript{2}, M Yu Gutkin\textsuperscript{1,3,4}

\textsuperscript{1}Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia
\textsuperscript{2}Ioffe Institute, RAS, St. Petersburg, Russia
\textsuperscript{3}Institute of Problems of Mechanical Engineering, RAS, St. Petersburg, Russia
\textsuperscript{4}ITMO University, St. Petersburg, Russia

E-mail: gudkinazhanna@mail.ru

Abstract. Results of Synchrotron Radiation (SR) microtomography of human dentin specimens subjected to compression tests are presented. A special attention is paid to cracks formed in the samples under compression. In all the samples, the dominant crack paths were observed to deviate and deflect around certain areas at or near to dentinal tubules. It is confirmed that secondary (satellite) cracks play an important role in dominant crack blunting. It is shown that microtomography is an effective method for investigation of the formation of satellite cracks and crack-tubule interactions over surface areas and across the whole depth of a dentin specimen. These processes can be visualized and evaluated using reconstructed 2D slices. A qualitative model for the formation of secondary cracks opening at stress concentrators that are dentinal tubules is suggested. It is shown that on the surface of the tubules, which are the closest to the tip of a mode II crack, the tensile circumferential stress can reach extremely high values that are more than enough for generation of secondary cracks.

1. Introduction

Dentin is a natural porous material with a hierarchical structure which includes at least seven scale levels. For example, the dentin tubules are microscopic quasi-cylindrical channels of 3–5 \(\mu m\) in diameter, which pass through dentin matrix and are spaced apart a distance of 10 \(\mu m\), whereas the characteristic diameter of nanoscopic collagen fibrilles, which compose the dentin matrix, is about 100 nm. For many decades, specific strength and toughness properties of dentin have been in the focus of extensive studies, however their origin is not clear so far. It is explained in part by the fact that no imaging technique can display at least several structural levels of dentin at the same time.

Among other important topics related to human dentin, there has been much interest in the processes of crack propagation in it. Research in this area has shown that the salient toughening mechanism is crack bridging by uncracked ligaments \[1–5\]. The so called ‘bridges’ of intact material were observed directly by optical and scanning electron (SEM) microscopies \[1, 2, 4\] and X-ray tomography \[1, 5\]. On the surface of a dentin specimen (treated with conditioning chemical agents) SEM, optical and atomic force microscopies show the openings of tubules. Improved image resolution achieved in SEM and micro-tomography techniques allows to observe microcracks in the vicinity of the main crack, which form deflections and branching in the crack wake. The evidence for tubule interactions with a growing microcrack were provided \[4\].
However, no mechanism has been proposed to explain how secondary (satellite) microcracks are formed and how a microcrack is attracted to a tubule.

In the present work, synchrotron phase sensitive tomography has been used to study the development of microcracks in dentin specimens tested under uniaxial compression. Based on the experimental results, we propose a model for the generation of secondary microcracks in the vicinity of the initial crack tip. The tensile circumferential stress over the surfaces of the tubules in the vicinity of the crack tip is evaluated and shown to be high enough to promote the generation of satellite cracks.

2. Experimental procedure

The experiments were made with dentin samples cut from the roots of caries free human teeth. The samples with 0.5 mm thickness \(h\) had length \(l\) and width \(w\) dimensions equal to: \(l = 5\) mm, \(w = 3\) mm, so the ratio \(d/h = 12\), where \(d = \sqrt{l^2 + w^2}\). Strength properties of dentin cuboids tested under uniaxial compression were shown to depend on the \(d/h\) ratio [3]. Specifically, in the compressed samples with the ratio of \(d/h\) equal to 10–12, cracks were detected, although the samples were not separated under the test until the compressive stress reached \(\sigma = P/(lw) = 840\) MPa. To the samples tested in this study, a compressive stress of \(\sigma = 370\) MPa was applied. The plastic deformation was 15%.

Post-test observations of crack paths inside a specimen were obtained using X-ray tomography. Nowadays two ways to implement X-ray tomography are available: conventional absorption and phase contrast modes. In the absorption mode, tomography has a serious limitation in the visualization of micrometre-size features present in the sample like holes, pores, inclusions, etc. Absorption decreases with decreasing a feature size. At the same time, the refraction of X-rays increases with respect to the feature size. In the light provided by a coherent SR source the increase comes to the point where the feature image is determined only by the phase. X-ray phase contrast allows visualization of tiny objects. However, in order to obtain real-space parameters of the object, it is necessary to solve the inverse problem. Up to now, no such solution has been attempted for dentin material.

Our objective was to investigate dentin specimens whose surfaces were treated with no chemical agents. The damaged layer that appeared during cutting was removed with fine-grain \((10\, \mu m)\) abrasive paper. The incoming beam, which was not limited to a selected coherent fraction, illuminated a large sample. Experiments were performed on the third generation SR source: Pohang Light Source, Pohang city, Korea. On 6c biomedical-imaging beamline a multipole wiggler port provides high intensity radiation with the lateral coherence length of \(31\, \mu m\) in the vertical direction. X-ray images were obtained with 25 keV monochromatic

![Figure 1. Left panel: rendered 3D-image of the dentin specimen. Beam direction is antiparallel to the \(x\) axis. Rotation axis is parallel to the \(z\) axis. The sample-to-detector distance is 5 cm. Detector is parallel to the \(yz\) plane. Right panel: reconstruction region size along the \(z\) axis is reduced from 702 to 181 \(\mu m\).](image)
radiation and converted into visible light images using a YAG:Ce scintillator crystal of 30 μm thickness. A magnified light image had an effective pixel size of 0.32 μm.

When the specimen is rather thick, there are many tubules in the beam path. The wave function of radiation is variable and a phase-contrast approach (see, e.g. [6,7]) is not applicable. In this study, microtomography is used to visualize the directions of tubules, rather than to evaluate the tubules themselves. Separated images from several tubules can be recorded until the image size of a tubule exceeds the distance between them. Conditions for observing the arrangement of tubules are met in the near field. When the specimen-to-detector distance is \( z = 5 \) cm, the diameter of the first Fresnel zone is \( D_1 = 3.2 \) μm. It is smaller than the diameter of a tubule \( D = 4 \) μm. With increasing the distance \( z \), the image size may exceed the intertubular spacing and tubule images overlap.

Samples were mounted on a rotation stage so that \( z = 5 \) cm and projections were recorded every 0.2°. Normalization and reconstruction of the projection images were obtained using Octopus V8.7. Visualization and 3D-rendering were performed with the aid of Amira V5.4.

3. Results
Rendered three-dimensional (3D) image of original dentin specimen before testing is shown in Fig. 1. In the left panel, a full field of view has the sizes 832, 832 and 702 μm along the axes \( x \), \( y \) and \( z \), respectively. In the right panel, the amount of contrasts in the reconstruction region is reduced by decreasing the size of the region along the \( z \) axis from 702 μm to 181 μm. The linear contrasts are observed to extend between the surfaces of the sample. They substantially deviate from the straight lines by which tubule directions are usually approximated (see, e.g., [1, 6, 7]). We believe that the linear contrasts show the directions of tubules. However, we note that these contrasts do not contain information about the tubules themselves.

After a compression test, cracks appear at the surface of the material and penetrate the entire volume of the sample. Scheme of uniaxial compression of the specimen (that has the \( d/h \) ratio of 12) is shown in Fig. 2. The compressive load \( P \) is parallel to the \( x \) axis. The 3D images display the main plane of the crack, whose detailed behavior in the interior of the specimen is examined using 2D reconstructed slices (tomograms).

On tomograms, tubules appear as dark spots and cracks appear as black and white lines (Fig. 3). Since in our experiments the tubules are inclined with respect to the axis of rotation in a random manner, they seem to be randomly distributed on a tomogram. In addition, their cross-sections have random shapes which are not at all like circular voids [6,7]. Moreover, in
Figure 3. X-ray tomograms computed for a dentin specimen containing cracks. Tubule cross-sections are represented with dark spots. Some tubules are highlighted with rounded shapes. (a) Dominant crack starts from the sample edge and splits into two components: 1 and 2. The crack paths deviate around certain areas with separation distances of the order of 10 µm or a multiple of 10 µm. (b) This tomogram locates 50 µm 'below' the one shown in (a). The crack 1 splits into several satellite cracks, as highlighted by white arrowheads for the locations of crack tips.

the vicinity of crack images the visibility of the tubules is poor because the cracks show always a brighter contrast. However, despite these obstacles and complications, the examination of the crack shapes suggests that the tubules are involved in the crack paths. On the one hand, the paths deviate around certain areas. In Fig. 3a both the upper 1 and lower 2 cracks deflect around the areas separated by a distance of 10 µm or a multiple of 10 µm. On the other hand, the value of 10 µm does not contradict the conventional tubule spacing in dentin, and does not disagree with SEM data [4]. We believe that the deflections of dominant cracks (Fig. 3a) occur around the tubules when the latter are intersected by propagating cracks.

Figure 3b represents the differences which occur with depth. The tomogram in Fig. 3b locates 50 µm below the one shown in (a). The depth values (from the edge of the view field of the detector) are given in micrometres in tomograms. One can see the initiation of secondary (satellite) cracks ahead of the crack 1. In other words, the formation of bridges of intact material impedes the opening of the crack 1. SEM observations of similar phenomenon performed on the dentin surface treated with chemical agents revealed, in addition, the difference between the tubules. Some tubules can cause the crack deflection, while others do not [4]. Microtomography suffers from some uncertainty caused by overlapping images. In the meantime, it is a method capable of non-destructive depth analysis of shapes and sizes of microcracks.

4. Model
Consider a qualitative theoretical model which explains, in the first approximation, the reasons for the formation of secondary (satellite) cracks in dentin under compression. Figure 4 illustrates the opening of a mode II crack in a dentin sample which is compressed across the average orientation of dentin tubules. The crack starts from the 'top' surface of the sample under a compressive stress $\sigma$ and propagates (until blocking) to a distance $L$ under the shear stress $\tau$ (Fig. 4a,b). The crack plane is parallel to dentin tubules and inclined to the sample surface. As a result of the crack opening, a step of height $B$ is formed on the surface.

To analyze the stressed state at the tip of the crack, it is convenient to use its dislocation model (Fig. 4c) which is a pile-up of $N$ edge dislocations with the Burgers vector $b$ [8]. The number $N$ of the dislocations in such a pile-up is determined by the values of the shear stress $\tau$ and the crack length $L$ [8]: $N = \pi(1 - \nu)L\tau/(Gb)$, where $\nu$ is the Poisson ratio, $G$ is the shear modulus, and $b = |b|$. In the case under consideration, the elastic moduli $\nu$ and $G$ are taken in the sense of overall effective elastic constants of a porous material with unidirectional packing
Figure 4. Sketch of mode II crack opening in a dentin sample under compression. (a, b) The crack starts from the 'top' surface of the sample under a compressive stress $\sigma$ and propagates to a distance $L$ under the shear stress $\tau$ in a plane parallel to the average orientation of dentin tubules and inclined to the sample surface; as a result, a step of height $B$ is formed on the surface. (c) The crack is modeled by the corresponding pile-up of $N$ edge dislocations with the Burgers vector $b$. (d) The pile-up of $N$ dislocations is modeled by a superdislocation with the Burgers vector $B = Nb$.

In the first approximation, the pile-up of $N$ dislocations can be modeled as an edge superdislocation (Fig. 4d) with the Burgers vector $B = Nb$ with magnitude $B = \pi(1 - \nu)L\tau/G$ (equal to the surface step of height $B$ shown in Fig. 4a). In this case, one can qualitatively

Figure 5. Model of a blocked initial mode II crack in the plane parallel to dentin tubules under remote shear stress $\tau$. The stress field in the vicinity of the crack tip is modeled by that of an edge superdislocation. Near the crack tip, the signs of acting normal and shear stresses are sketched. The secondary cracks are opened at dentin tubules, where the tensile stresses reach their maximum values, and propagate in directions different from the direction of the initial crack growth.
image the stressed state around the crack tip with using the well-known stress field of an edge dislocation in an elastically isotropic infinite solid [8]. In doing so, we sketch the signs of acting normal and shear stresses near the crack tip in Fig. 5. Based on this sketch, we suppose that the secondary cracks are opened at stress concentrators that are dentin tubules, where the tensile stresses reach their maximum values. Therefore, the generation of secondary cracks is supposed at the dentin tubules just near the initial crack tip, in the areas ‘ahead’ and ‘under’ the superdislocation (Fig. 5).

To provide a deeper consideration of secondary crack generation, we have used the solution of the boundary-value problem in the theory of elasticity for a straight edge dislocation placed near a cylindrical void [10]. In this approach, the edge dislocation models the crack tip, while the void is a model of an individual dentin tubule. It is worth noting that we have not taken into account the effect of surrounding dentin tubules on the stress distribution around the probe dentin tubule, which is correctly enough when the average distance between the dentin tubules is much larger than their diameters (Figs. 3 show that this is true for our samples of dentin). Thus, using the solution [10], we have calculated the distribution of tensile circumferential stress over the surfaces of the tubules in the vicinity of the crack tip. For some exemplary tubules, which are the closest to the crack tip and are distant by 7 μm from it, the tensile stress has been shown to reach extremely high values about of 1.5 GPa (for the remote compressive stress $\sigma = 370$ MPa) that seems to be more than enough for generation of secondary cracks in dentin sample with characteristic Young modulus about of 2 GPa (in the case of compression test for $d/h = 12$, see [11] for details).

5. Summary
We used the results SR microtomography of human dentin specimens subjected to compression tests to shed some light onto the crack evolution in dentin under compression. We observed that the dominant crack paths deviate and deflect around certain areas at or near to dentinal tubules and showed that SR microtomography is an effective method for investigation of the formation of satellite cracks and crack-tubule interactions. We also developed a qualitative model for the formation of secondary cracks opening at stress concentrators that are dentinal tubules, and showed that on the surface of the tubules closest to the dominant mode II crack tip, the tensile circumferential stress can reach extremely high values, thus providing generation of secondary cracks.

Acknowledgements
The authors are thankful to Dr. D. V. Zaytsev for providing the samples of human dentin used in this work. M. Yu. G. thanks the Russian Foundation of Basic Research for the support (grant No. 18-38-20097).

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