

Structure and Deformation Behavior of Human Dentin

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Abstract

The relationship between structure and stress accommodation mechanisms (deformation and fracture) of human dentin on macro-, micro- and nano- scales is discussed. Dentin is the hard basis of human teeth with complicated hierarchically organized structure, which is attested as a natural composite consisted of a bioorganic matrix armed by collagen fibers and apatite crystallites. Dentin exhibits the unique strength properties. On the macroscopic level, under tensile load, it behaves like a brittle solid, and like a viscoelastic one in the case of compression. At the same time, on the microscopic scale cracks in dentin grow in a viscoelastic manner under tensile loading. Structure, mechanical properties and crack growth of human dentin on macro-, micro- and nano- scales, including TEM study, are considered in detail. It was shown that a brittle response under tension is the macroscopic feature of dentin caused by dentin channels, while viscoelasticity is its intrinsic property.

Keywords: Human dentin; Structure; Deformation; Fracture; Crack

1. INTRODUCTION

Human teeth are the part of the human maxillofacial apparatus that serve mainly for grinding food. Dentin is the hard basis of a tooth, which possesses complicated hierarchically organized structure [1]. This hard tissue contains 70% of bioorganic compounds, 20% of inorganic compounds and 10% of water [2]. Its deformation behavior exhibits many unique features in comparison with some natural and synthetic materials [3]. For example, at room temperature human dentin demonstrates a brittle deformation behavior under tension at the macroscopic level, while it behaves as a viscoelastic material under compression in the same conditions. At the same time, on the microscopic scale cracks in dentin grow by a viscoelastic manner under tensile loading. For better understanding of this effect, an information on the fracture behavior of human dentin on the next to microscopic level, that is nanoscale level, is required. Majority of the works on deformation and fracture of dentin was carried out on the macroscopic and microscopic scales [4]. There were only few studies on the structure and accommodation of stresses in dentin at the nano level in the literature [5]. The

complicated technique of dentin sample preparation for transmission electron microscope (TEM) is the main cause for this abnormal situation. This paper is devoted to the discussion of structure of human dentin on macro-, micro- and nanoscales and its relationship with its deformation behavior for every level.

2. HIERARCHY OF STRUCTURE IN HUMAN DENTIN

There are two parts in a human tooth [6]: a crown and a root (Fig. 1). Crown rises above a gum, while root seats in a cell of jawbone. Layer of tooth enamel, which is the hardest tissue of the human body, covers crown for protection from mechanical and corrosion damages. Tooth cement covers root that provides a ligament of a tooth with a gum. The main volume of a tooth consists of dentin. There is a pulp chamber in the middle of the tooth, which passes into a narrow channel ending with a hole at the top of the root or root channel. Pulp, which consists of a plexus of nerve fibers and blood vessels, is situated in this cavity. The pulp of the tooth is connected to the tissues of the jaw by a neurovascular bundle that passes through a

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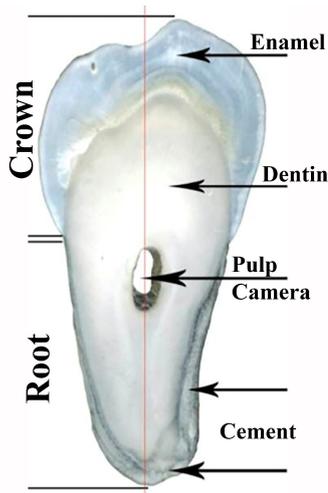


Fig. 1. Cross-section of human premolar (optical microscope in the reflection mode).

hole in the apex of the tooth root [7]. Chemical composition and structure of dentin in different parts of a tooth is also different. For example, the water content in dentin in vicinity of dentin-enamel junction is about 1%, while it is about 22% in the near-pulp dentin. It means that hydration of dentin in different places of a tooth is different and can vary by up to 20 times [7].

The main morphological feature of dentin on the microscopic scale is dentin channels, where the dentin fluid circulates. Chemical composition of the dentin fluid is similar to the human oral fluid [2]. Dentin channels are surrounded by calcium hydroxyapatite crystallites (20–40 nm in diameter and 0.5–1 nm in thickness) and collagen fibers (0.5–1 nm in diameter), which reinforced a bioorganic dentin matrix. This area is determined as inter-tubular dentin. The channels are equidistantly spaced from one to another (Figs. 2 and 3). The diameter of the channels varies from 5 μm near pulp camera and root canals, to 1 μm in vicinity of both dentin-enamel junction and dentin-cement junction. Dentin channel walls are rough due to the protrusion of hydroxyapatite crystals and

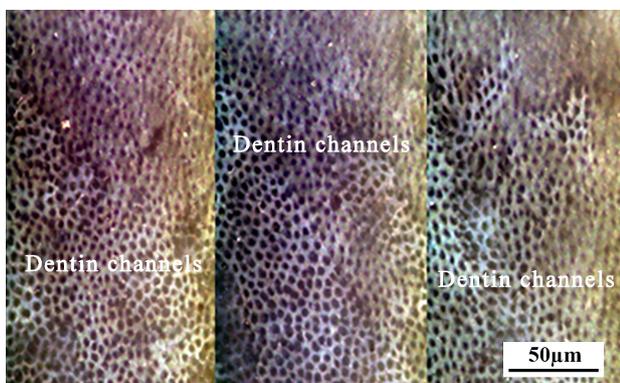


Fig. 2. Microstructure of human dentin: distribution of the dentin channels in the cross-section that is normal to the axis of dentin channels (optical microscope in the transmission mode).

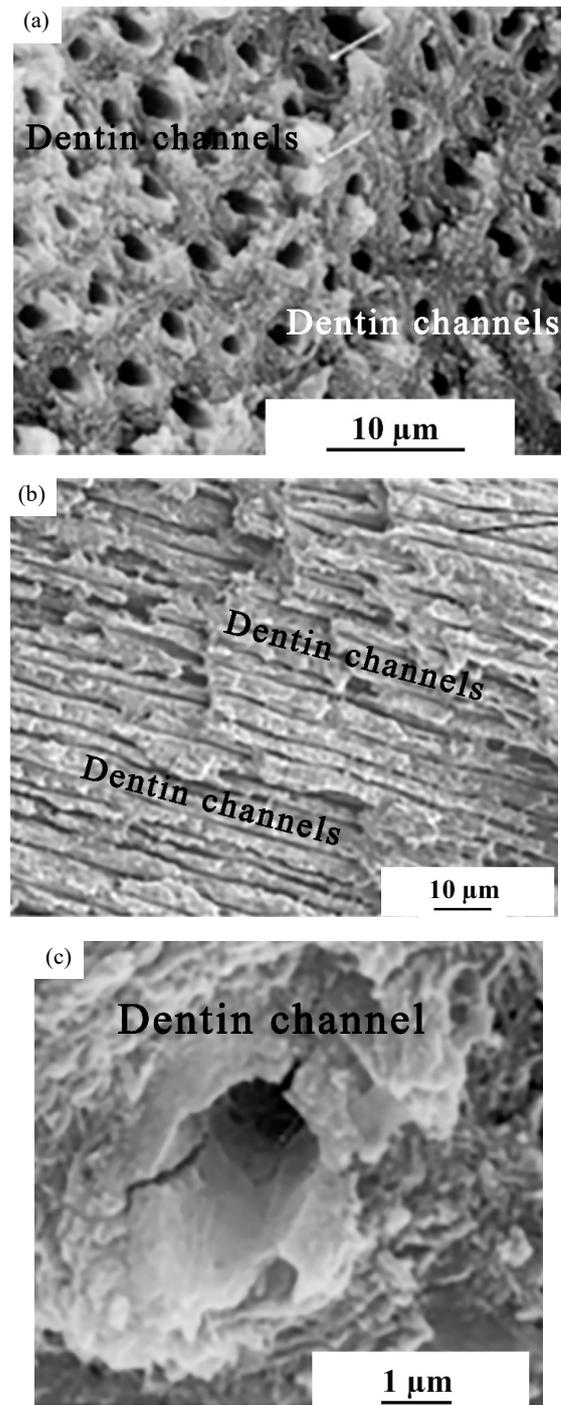


Fig. 3. Microstructure of human dentin: (a) cross-section that is normal to the axis of dentin channels; (b) cross-section that is parallel to the axis of dentin channels; (c) dentin channel (SEM images). Adapted from Ref. [5].

collagen fibers into their lumen and a large number of lateral branches and microtubes, which serve as connections between neighboring channels [8]. X-ray tomography was used to examine the distribution of dentin channels in a tooth [9]. It allowed to describe the structure of dentin matrix and to perform its 3D reconstruction. The images taken from Ref. [8] show three-dimensional grid of dentin channels (Fig. 4).

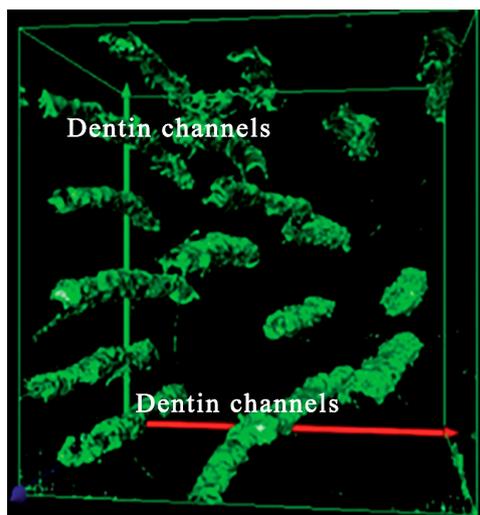


Fig. 4. 3D computer tomographical reconstruction of human dentin structure: the distribution of dentin channels. Adapted from Ref. [8].

Studies of intact and sclerosed dentin by patients aged 25–30 years old and 65–80 years old, respectively, were performed using the TEM method at the nanoscale [10]. Samples were prepared by the microtome technique, and final thinning was performed by ion etching. Areas of dentin near the top of the tooth and the border between the root and the crown were studied. Intertubular crystals were needle-shaped in both intact and sclerosed dentin. Although the needle-shaped crystallites were mostly straight, curved crystals were also seen in some cases (Fig. 5a). Differences in the crystal structure between these types of dentins were not revealed. Visualization of the structure of the intertubular dentin showed that it contains lamellar crystals of hydroxyapatite (Fig. 5b). Intertubular hydroxyapatite crystals in sclerosed dentin were smaller than crystals in intact dentin, while there were no differences in their chemical structure. The intratubular hydroxyapatite crystals were larger than the intertubular ones, but there were no differences in chemical composition (Fig. 6).

Dentin of intact teeth of teenage patients (12–16 years old) and mature (30–40 years old) patients was examined. Thin foils for TEM from dentin were prepared by a chemical thinning in concentrated H_3PO_4 . According to TEM study, both amorphous and ultrafine crystalline phases are present in teenage and mature dentin. The amorphous phase is dominant in teenage dentin, while the ultrafine phase becomes dominant in mature dentin. It can be concluded that initially calcium hydroxyapatite of human dentin was in an amorphous state (in teenage period), but later it begins crystallizing, and upon reaching adulthood, calcium hydroxyapatite completely transforms into an ultrafine crystalline state [11].

TEM images of the teenage dentin structure of patients aged 13–14 years old were obtained in the "absorption

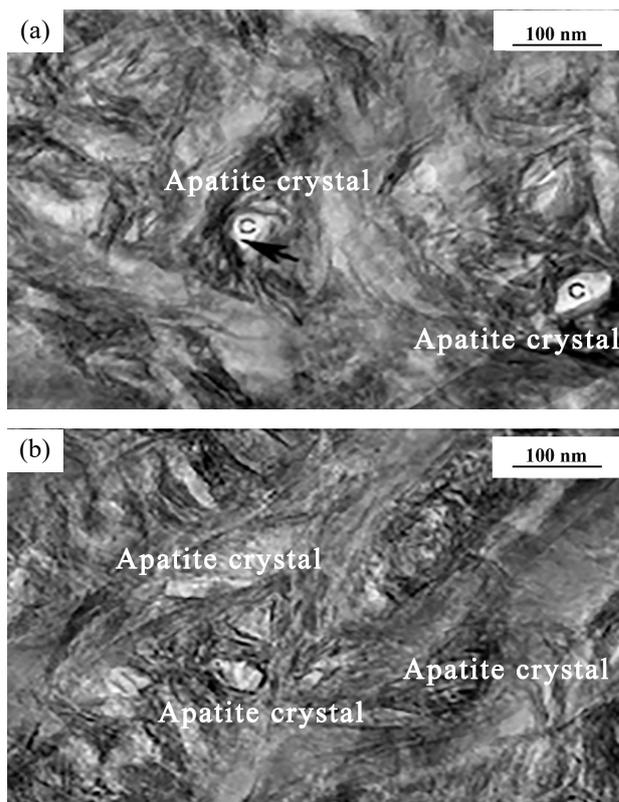


Fig. 5. Structure of intertubular dentin in the nano- scale (TEM images): (a) transparent dentin; (b) normal dentin. Adapted from Ref. [10].

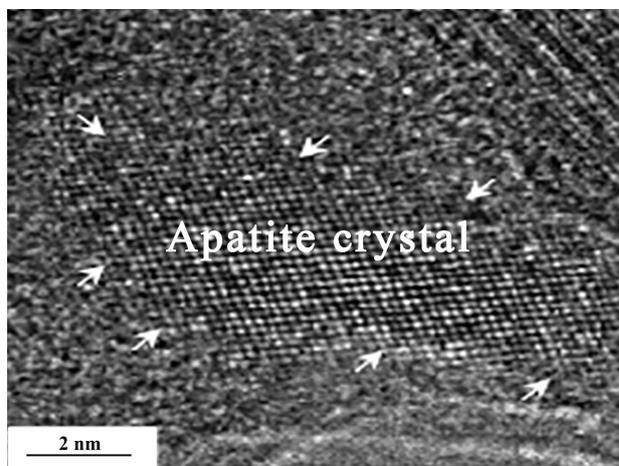


Fig. 6. Structure of sclerosed mature dentin in the nanoscale. The arrows show intertubular mineral crystal (HREM image). Adapted from Ref. [10].

contrast" mode and are therefore unsharp even after digital processing. Diffuse halo is observed in the electron diffraction patterns obtained from thin foils, which indicates the amorphous state of the dentin matrix. It has been shown that, regardless of the location in the tooth, teenage dentin has a layered morphology: the foil consists of a stack of layers 50–100 nm thick, oriented perpendicular to the main axis of the tooth (Fig. 7a). The layered structure of dentin is explained by the peculiarities of the process of

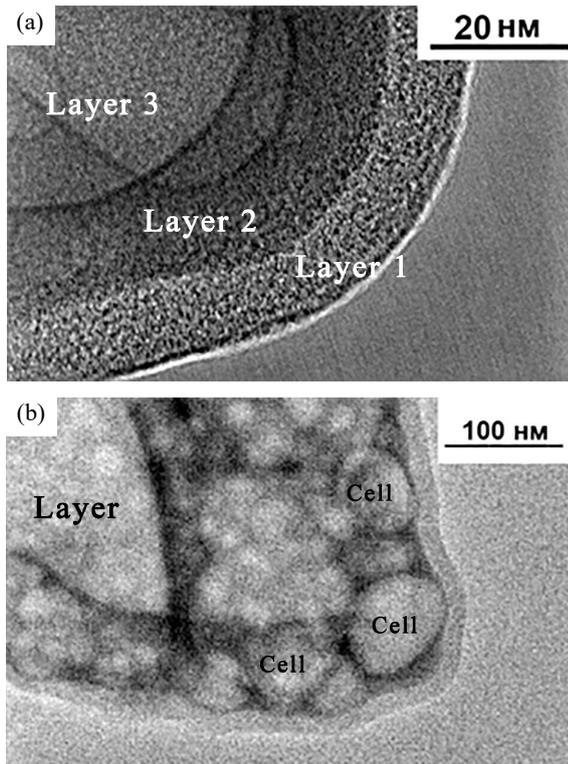


Fig. 7. Structure of the teenage dentin (14 years old) in the nanoscale (TEM images): (a) layered morphology; (b) cellular structure.

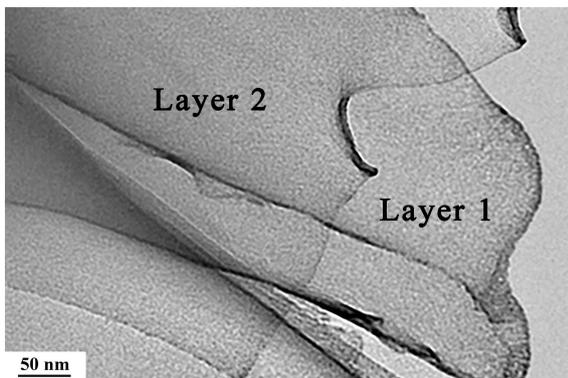


Fig. 8. Layered structure of the teenage dentin (18 years old) in the nanoscale (TEM images). Adapted from Ref. [22].

dentinogenesis in the human body. Light concentric cells 50–100 nm in diameter are observed in the layers. Between the cells there are dark boundaries 10–20 nm wide, within which no fine structure was found (Fig. 7b).

Teenage dentin of patients aged 16–18 years old also has a layered morphology (Fig. 8). It consists of thin layers 50–100 nm thick, which are perpendicular to the main axis of the tooth. The layers contain concentric cells 40–100 nm in diameter (Fig. 9). However, in contrast to the dentin of patients aged 13–14 years old, dark fibers with a thickness of ~5 nm appeared within the boundaries of the cells of the dentin of patients aged 16–18 years old, which were certified as collagen fibers (Fig. 9). No hydroxyapatite crystals

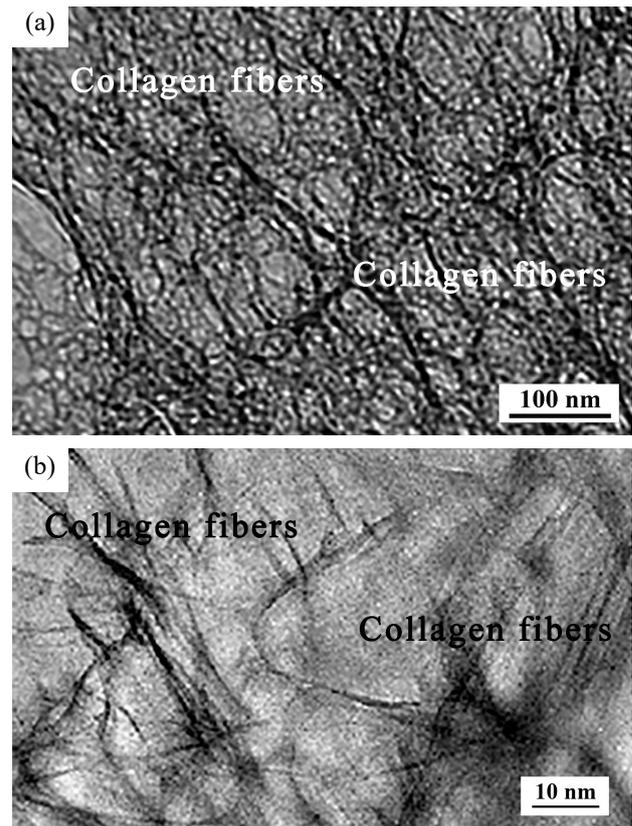


Fig. 9. Structure of the teenage dentin (16 years old) in the nanoscale (TEM images): (a) cellular structure formed by collagen fibers; (b) collagen fibers. Adapted from Ref. [22].

were found in thin foils as well as in the dentin of patients aged 13–14 years old. Perhaps, they dissolved in acid during thin foil preparation.

TEM study of mature dentin has shown that dark inclusions with a complex shape and size were visible on a light gray field, which varied from 10 nm to 100 nm (Fig. 10a). No traces of collagen fibers were observed. Both diffuse halos and diffractive spots, which tended to coalesce in rings, were clearly visible (Fig. 10b). Dark-field imaging evaluation (Fig. 10c) showed that some inclusions in mature dentin were 10–100 nm in size.

Thus, it may be concluded that human dentin represents a bioorganic matrix reinforced with collagen fibers and filled by hydroxyapatite crystallites, which elastic and plastic properties are similar to those of filled polymers, such as rubber, but its strength is close to a biomineral — human tooth enamel [12,13]. The source of such behavior is the bioorganic component of human dentin. Collagen fibers provide elasticity and plasticity to dentin [14] and calcium hydroxyapatite nanocrystals in the bioorganic matrix serve as a filler that strengthens dentin. Hence, a brittle response of dentin under tension should be its macroscopic feature, while viscoelasticity is the intrinsic property of human dentin.

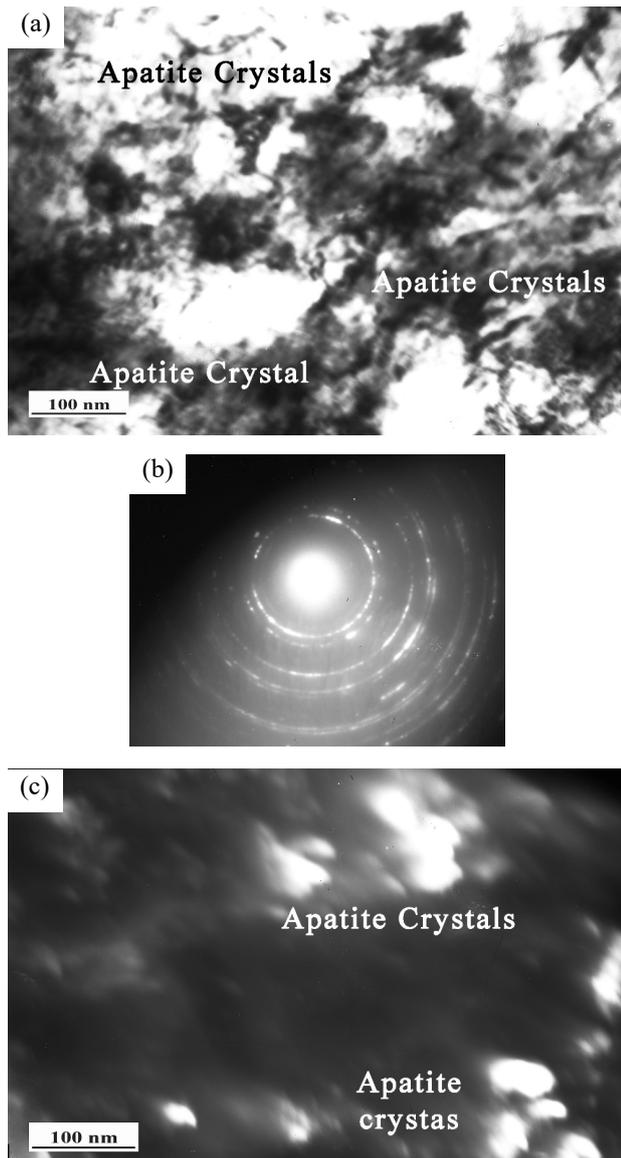


Fig. 10. Structure of mature dentin in the nano- scale (TEM images): (a) bright field; (b) electron diffraction; (c) dark field. Adapted from Ref. [11].

3. MECHANICAL PROPERTIES OF HUMAN DENTIN

The mechanical properties of human dentin under various types of loading are described in detail in the literature [4]. Zaitsev et al. have shown that the human dentin under compression behaves like an elastic-plastic solid body [4]. Deformation curves of dentin under compression are shown in Fig. 11a. The compression curve of dentin can be divided into two sections: linear (at the initial stage of testing up to stresses ~ 60 MPa and strains $\sim 3.5\%$) and, further until the moment of failure, non-linear. The dentin specimens showed the ability to both large reversible/elastic and significant irreversible deformation. It is assumed that the sample is deformed reversibly/elastically in the linear section, and irreversibly in the nonlinear section.

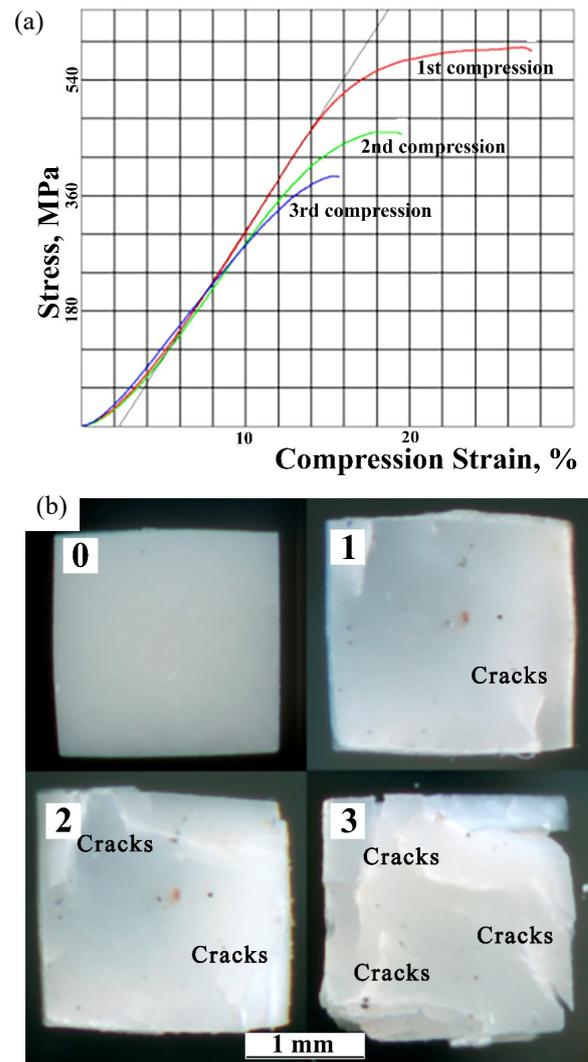


Fig. 11. Deformation behavior of human dentin under uniaxial compression: (a) stress-strain curves; (b) working surface of the sample (0 – in undeformed state, 1 – after first compression; 2 – after second compression; 3 – after third compression). Adapted from Ref. [4].

Dentin sample did not fail and deformed elastically, although the amount of plastic deformation decreased with each test (Fig. 11b). Therefore, dentin is capable of multiple loading cycles under compression load, that is, it behaves like a viscoelastic material, for example, as an elastomer. Thus, human dentin is able to function without failure and with the preservation of elastic properties even in the presence of cracks in samples, which is typical for a viscoelastic material.

Deformation curves of human dentin under diametrical compression, when tensile loading is applied to a sample, in different environments (air, water, 18% water solution of NaCl, liquid nitrogen) are shown in Fig. 12 [15]. A straight line can approximate the curves for room temperature. This fact may be interpreted as a feature of a brittle deformation behavior; however, the value of strain (4–7%) is closer to a viscoelastic behavior than to a brittle response. The tensile

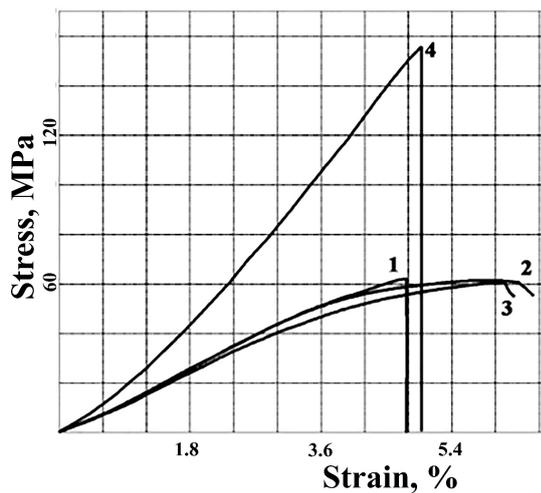


Fig. 12. Stress-strain curves of human dentin under diametrical compression in various media: 1 – in air, 2 – in water, 3 – in 18% water solution of NaCl, 4 – in liquid nitrogen (77K). Adapted from Ref. [17].

strength of dentin increased from 60 MPa when testing in air and water at room temperature to 150 MPa when testing in liquid nitrogen. Deformation prior to the failure of the sample under testing in air and in liquid nitrogen is the same (~5%), while it increases in water environments (~7%). At room temperature, dentin samples change the shape from circle to ellipse. This fact points to some inelastic deformation that occurs in human dentin. As a result, three cracks appear on the back surface of sample in the places of maximal tensile stress (Fig. 13a).

The dentin sample tested in liquid nitrogen fails on two equal semicircle parts along the line of application of the compressive loading without plastic response (Fig. 13b). The linear slope of the deformation curve and the failure of the sample due to the growth of a single crack allows to conclude that dentin at $-196\text{ }^{\circ}\text{C}$ behaves like a brittle material [16,17]. However, the value of strain (~5%) does not allow to accept this statement. An increase in the strength of dentin (by about 2.5 times) is a characteristic feature of testing a polymer material at low temperatures. It seems that it is a viscoelastic behavior of a filled polymer. The absence of plasticity in dentin in liquid nitrogen means that in this case plasticity is realized only due to the elasticity of bioorganic component of the dentin matrix. When dentin tested in air, its plasticity can be realized both due to the elasticity of bioorganic component and porosity of the dentin matrix, namely dentin channels. Indeed, plasticity of dentin becomes close to zero under testing in water despite the increase of strain up to 6–7% due to the growth of its elasticity only. The filling of dentin channels by water is the cause of this effect.

Analysis of deformation behavior of human dentin under compressive and tensile loadings has shown that human dentin exhibits viscoelastic behavior independently

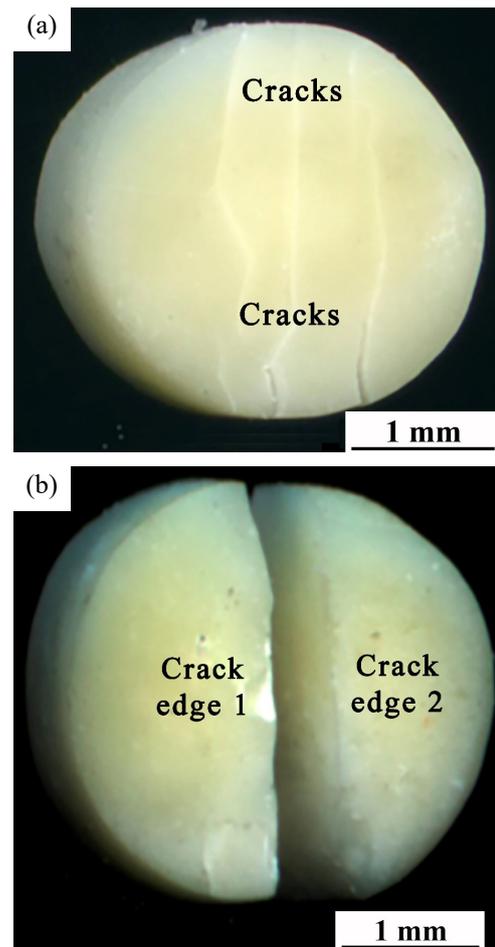


Fig. 13. Working surface of human dentin samples after diametrical compression: (a) in air at room temperature; (b) in liquid nitrogen (77K). Adapted from Ref. [4].

from temperature and environment of testing. Considering the structure of this hard tissue, including its chemical content, it may be concluded that such behavior is the intrinsic response of dentin on the application of mechanical loading. On the other hand, the brittle or rather quasibrittle response to tensile loading is the macroscopic feature of human dentin.

4. CRACK GROWTH IN HUMAN DENTIN

R.O. Ritchie with co-workers have done considerable contribution to the understanding of fracture mechanisms of dentin [3]. Detailed description of crack growth in bulk samples was carried out in their works. Observations have shown that for all orientations of the dentinal channels in samples, cracks appear in the plane of the notch and the value of crack resistance is maximum when a crack propagates parallel to the dentinal channels. However, it is minimal, if a crack grows perpendicularly to the channels. When the cracks deviate from the plane of maximum tensile stresses, the stresses at the crack tip decrease, providing work hardening. Features of the dentin microstructure

can cause deviation of a crack from the initial growth direction, for example, a crack can change its trajectory when interacting with hydroxyapatite crystals [18]. With parallel orientation of the dentinal channels, there were practically no deviations of the crack from rectilinear propagation. It is assumed that the hardening with this orientation is negligible, which is also true for perpendicular orientation. The mechanism mentioned above assumes that the crack deviations are quite small $\sim 20 \mu\text{m}$. The largest contribution of deviations to stress reduction occurs with a perpendicular orientation; however, this statement was not confirmed by macroscopic strength measurements. In the case when the main crack is connected with small cracks at the tip, forming undamaged areas, the formation of bridges occurred (Fig. 14a,b). The formation of bridges between the edges of the crack is inherent in materials such as fiber composites. Observations of crack growth showed the possibility of bridge formation by collagen fibers (Fig. 14c). This mechanism is observed only with parallel orientation of the dentinal canals. The formation of microcracks in the damage zone in front of the crack tip often leads to a decrease in the strength of the material in this area and screening of the crack tip and, consequently, to hardening of the material [19,20].

Thus, dentin can be considered as a fiber-reinforced composite, with a brittle matrix of intertubular dentin reinforced with dentinal tubes. However, such a representation assumes that the strength of the material is maximum for cracks propagating perpendicular to the channels, while the experiment shows the exact opposite result. This contradiction and the fracture toughness values clearly suggest that the channels do not directly affect the strength of the material. Based on this, it can be concluded that the strength of dentin depends on the orientation of elastic collagen fibers and the formation of undamaged areas (bridges) between the edges of the crack.

Ritchie's conclusion sounds logical and can be confirmed in many experiments, but their supposition on a brittleness of dentin bioorganic matrix requires a proof and discussion. Indeed, it is unusual that a viscoelastic media behaves like a brittle solid at least on the microscopic scale. Rather, the brittle-like behavior of dentin under tensile loading in the macroscopic scale could be its property caused by the dentin channels.

The possible scenario of crack growth in dentin can be reduced to the following: in front of the main crack tip, there are small satellite cracks, the junction with which leads to an increase in the length of the main crack. Such a mechanism implies that there is a developed plastic zone in front of the main crack tip, comparable in size to the length of the crack, but experimental confirmation of its existence in dentin was not obtained in the Ritchie's works. It seems to be that described above mechanism of

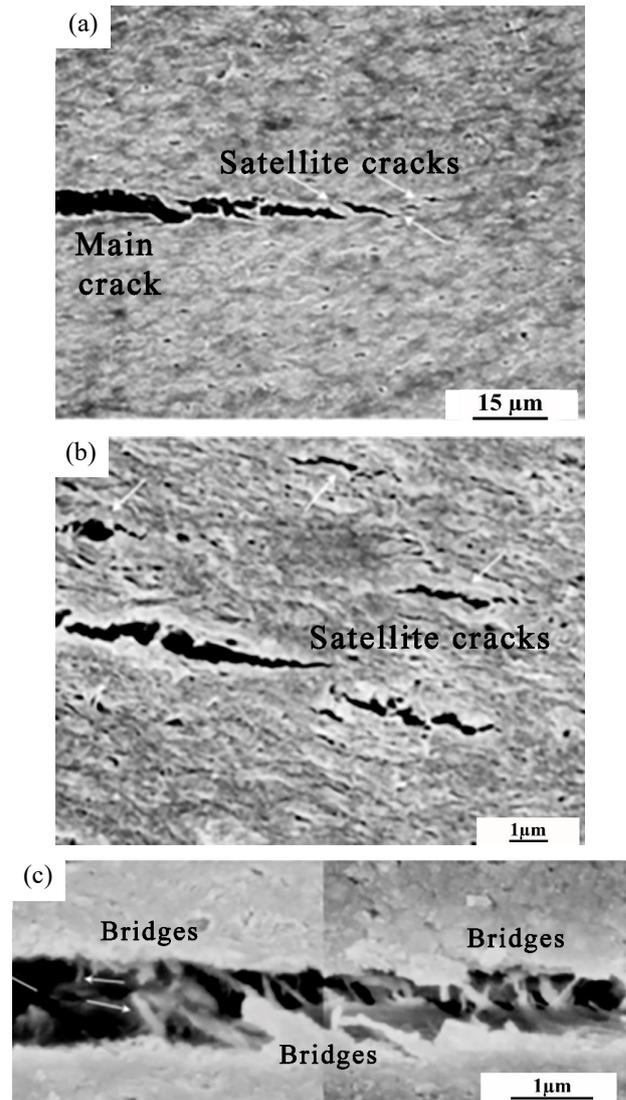


Fig. 14. Cracks in the bulk samples of human dentin (SEM images): (a) cracks are located in front of main crack; (b) cracks are located in front of main crack; (c) edges of crack contain collagen fibers (indicated by white arrows). Adapted from Ref. [3].

crack growth in dentin turned out to be close to the fracture mechanism of ductile metals [21]. Therefore, proposed model of crack propagation in dentin should be similar to the mechanism of crack propagation in ductile metal, although the mechanical properties of dentin described in the literature differ from those of metal. Therefore, such an analogy seems to be non-obvious and requires a detailed justification. For example, direct observation of the plastic zone in front of the main crack tip can serve as a proof.

Plastic zone containing satellite cracks in front of the main crack in human dentin was discovered by Zaitsev et al. in thin samples of human dentin [21]. Direct observations in transmission optical microscope have shown that satellite cracks develop in a long narrow thinned area of the dentin in front of the main crack tip, which was attested as a plastic zone (Fig. 15). Cracks in thin samples were

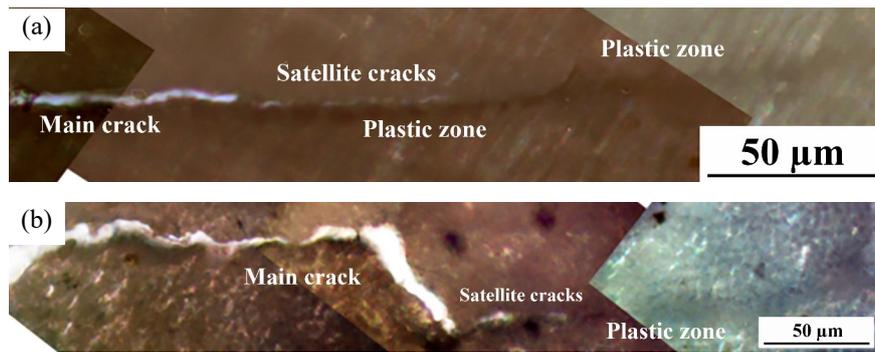


Fig. 15. Cracks with the plastic zone ahead in thin samples of human dentin (optical microscope images in the transmission mode): (a) dentin channel lay in the plane of the image; (b) dentin channel lay perpendicularly to the plane of the image. Adapted from Ref. [4].

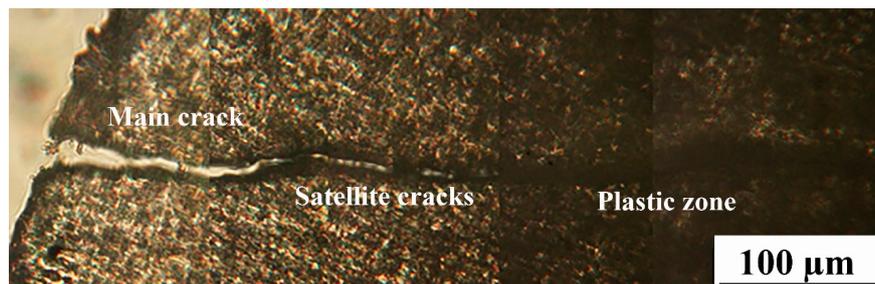


Fig. 16. Main crack with satellite cracks in the plastic zone ahead in thin samples of human dentin (optical microscope images in the transmission mode).

wedge-shaped with uneven edges, and their opening angle did not exceed 5° . A narrow dark strip is located in front of the top of the main crack (Fig. 16). Since the strip lies on the crack trajectory, it can be considered as the most likely place for the growth of the main crack. The cause of darkening may be a change in the conditions for the passage of light through the material, caused by the action of disjoining stresses. The trajectory of cracks in thin dentin samples did not depend on the orientation of the dentin channels, and the geometry of their tops was close to elliptical. Near the top of the crack, between the edges, there are areas of not destroyed dentin, which can be certified, according to Ritchie, as bridges [20]. Based on the data obtained, it can be concluded that at the micro level, dentin is destroyed similarly to a viscoelastic material, such as polyethylene film or rubber.

The study of cracks in the dentin in TEM at magnifications of the order of $\times 10K$ showed [11] that in front of their tips there was a thinning of the material and the formation of satellite cracks (Fig. 17a). The study of the dangerous crack tip area at high magnifications ($\times 100K$) confirmed that the edges of the main and satellite cracks were uneven, their tips were blunt, and the dentin matrix thinned in front of them, as the transparency of the material changed (Fig. 17b).

Tiny cracks in thin foils for TEM arose in places of localized accumulation of irreversible deformation, i.e., bends that appear during sample preparation, when tensile

stresses occur at the edges of the foil. After thinning of samples, the edges of thin foils contained numerous nano-scale tears, which were stress concentrators. During washing in water, as well as during installation in the microscope holder, the uneven edges of the foils were subjected to alternating loads producing nanocracks in places of stress concentration. Indeed, notch-like defects occurred

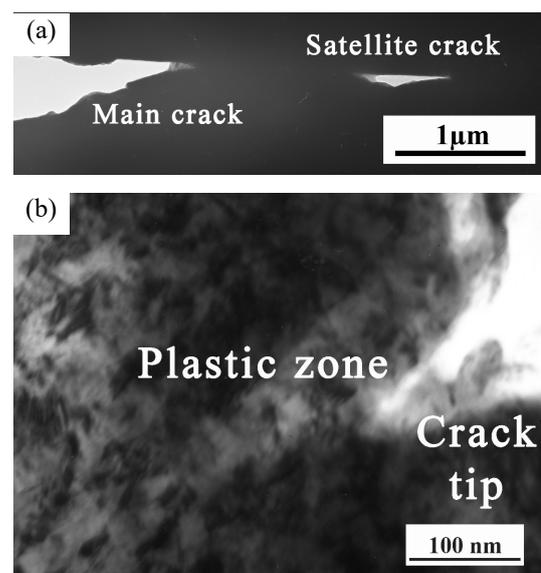


Fig. 17. Crack in thin foil of mature human dentin (TEM images): (a) satellite crack ahead main crack; (b) structure of dentin in main crack tip. Adapted from Ref. [11].

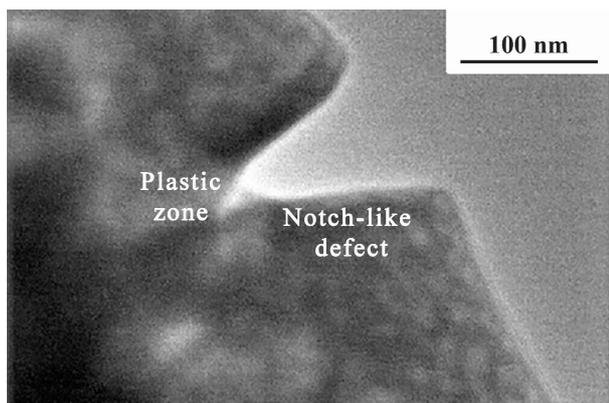


Fig. 18. Crack in thin foil of teenage dentin (14 years old) in the nanoscale (TEM images).

in the layer of adolescent dentin of patients aged 13–14 years old at the edges of the foils, but never spread to adjacent layers. They had a wedge-like shape with apex angles of 20–30°. In front of them, there was a thinning of the dentin, which indicated a significant irreversible deformation in this place (Fig. 18).

Cracks in the teenage dentin of patients aged 16–18 years old [22] also appeared and developed in the single layer of dentin (Fig. 19a). In this case, the adjacent layers had an ellipsoidal profile, which indicated a significant irreversible deformation in this section of the foil. The cracks had a wedge-shaped profile with angles at the apex of 10–15°, which is 2–3 times less than the angle at the apex of the notch in the dentin of patients 13–14 years old (Fig. 18). A narrow light area is observed in front of the crack tip (Fig. 19b). A possible reason for its formation is the thinning of dentin in the process of irreversible deformation, which is typical for plastic viscoelastic materials. In the undamaged layer, satellite porous cracks are observed located in front of the tip of the main crack.

Based on the data obtained, it can be concluded that the growth of cracks in thin foils of adolescent dentin at the nanoscale is similar to the growth of cracks in thin foils

of plastic metals and viscoelastic polymer films. That is, at the nanoscale, adolescent dentin is destroyed as a viscoelastic material. Features of the destruction of dentin are determined by its structure, for example, the angle at the tip of a crack depends on whether a network of collagen fibers has formed in the dentin matrix or not. Appearance of cracks in thin dentine specimens is preceded by localized accumulation of irreversible deformation near stress concentrators, which are considered as nuclei or places of the most probable occurrence of cracks [23]. The subsequent application of a tensile load to this area leads to the formation of a crack and, at the same time, to an intense thinning of the material in front of its tip. Thinning occurs in a narrow region, the morphology of which is determined by the geometry of the applied load. In the thinned region or plastic zone, satellite porous cracks appear which tend to merge with the main crack. Since dentin is a viscoelastic medium and behaves like a filled viscoelastic polymer [23], the mechanism of its destruction should be similar to the mechanisms of crack growth in ductile metals and viscoelastic polymers.

5. CONCLUSION

It was shown that under tension in air on a macroscopic scale, human dentin exhibits brittle behavior, while water causes its plastification and changing its type of deformation behavior from quasibrittle to viscoelastic. On a microscopic scale, dentin exhibits viscoelastic behavior, both in air and in water. The study of the failure of thin samples of dentin at the microlevel showed that in front of the tip of a dangerous crack there is a plastic zone, which is a section of thinned material where satellite porous cracks develop, as it occurs in viscoelastic solid. The TEM study showed that the viscoelastic nature of crack growth in dentin is also reproduced at the nanoscale.

The model of crack growth in dentin was based on the scheme of crack evolution in a ductile metal, which is

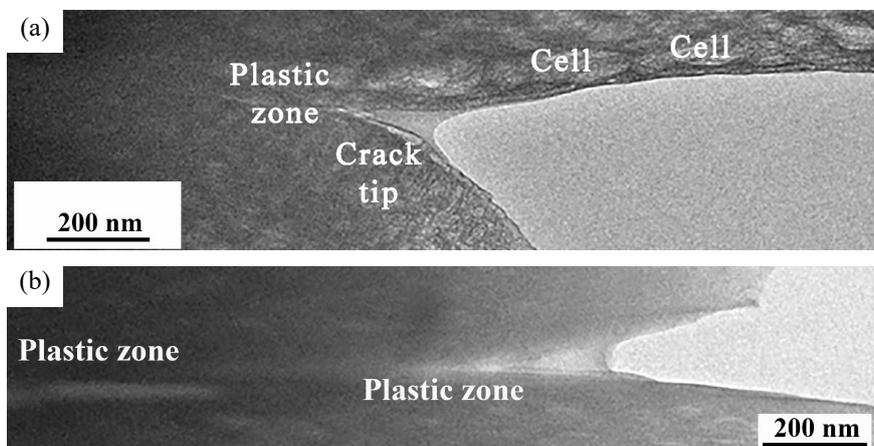


Fig. 19. Cracks in thin foils of teenage dentin (16 years old) in the nanoscale (TEM images): (a) crack with an area of localized accumulation of deformation ahead; (b) crack with satellite cracks ahead [22].

described in detail in the literature [24]. A dangerous crack in a metal occurs in the area of localized accumulation of deformation and, as a defect — stress concentrator — itself becomes a place of localization of deformation, which manifests itself in the form of a plastic zone in front of its tip [25]. The material in the plastic zone is intensively thinned, due to which pores appear there, which tend to merge with each other, which leads to the growth of the main crack. This fracture mechanism is observed on both micro- and nanoscales.

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Взаимосвязь структуры и деформационного поведения дентина зубов человека

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Аннотация. Обсуждается взаимосвязь структуры с механизмами аккомодации механических напряжений (деформация и разрушение) в дентине зубов человека на макро-, микро- и наномасштабах. Дентин представляет собой твердую основу зуба со сложной иерархически организованной структурой, который аттестован как природный композит, состоящий из биоорганической матрицы, упрочненной волокнами коллагена и кристаллитами апатита кальция. Дентин демонстрирует уникальные прочностные свойства. При растягивающей нагрузке он ведет себя как хрупкое твердое тело на макроскопическом уровне, а при сжатии — как вязкоупругий материал. При этом в микроскопическом масштабе трещины в дентине растут вязкоупругим образом при растягивающей нагрузке. Подробно рассмотрены структура, механические свойства и рост трещин дентина человека в макро-, микро- и наномасштабах, включая исследование с помощью просвечивающей электронной микроскопии. Было показано, что хрупкая реакция на растяжение является макроскопической характеристикой дентина, обусловленной дентинными каналами, а вязкоупругость является его внутренним свойством.

Ключевые слова: дентин зубов человека; структура; деформация; разрушение; трещины