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Short communication

Deformation behavior of human dentin in liquid nitrogen: A diametral compression test

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ABSTRACT

Contribution of the collagen fibers into the plasticity of human dentin is considered. Mechanical testing of dentin at low temperature allows excluding the plastic response of its organic matrix. Therefore, deformation and fracture behavior of the dentin samples under diametral compression at room temperature and liquid nitrogen temperature are compared. At 77 K dentin behaves like almost brittle material: it is deformed exclusively in the elastic regime and it fails due to growth of the sole crack. On the contrary, dentin demonstrates the ductile response at 300 K. There are both elastic and plastic contributions in the deformation of dentin samples. Multiple cracking and crack tip blunting precede the failure of samples. Organic phase plays an important role in fracture of dentin: plasticity of the collagen fibers could inhibit the crack growth.

1. Introduction

Measuring of mechanical properties of a brittle solid under tension by pulling apart of the grips in testing machine is connected with experimental inconvenient that is caused by the failure of sample in the grips. There are some alternative methods for estimating the tensile strength of materials, such as bending, indentation and diametral compression testing, in the literature [1]. The last of them, where the sample having tablet shape is being diametrally compressed by punches of testing machine from the opposite ends (Fig. 1a). Shear force at the points of contact of punches with the sample induces the pure tensile stress in diametral plane of the tablet sample, where fracture occurs. Such technique could be successfully applied to the testing of small-sized samples [2]. Therefore, it can be used for examination of mechanical properties of the hard tissues of living organisms, where the volume of materials is limited [2,3,4].

In the last time, diametral compression was used for testing the dental materials [5,6]. The lifetime of restoration of human tooth is mainly determined by the mechanical compatibility of restorative material with its hard tissues. There are small data on the deformation behavior of human dentin under this deformation scheme in a literature. It was reported that the ultimate tensile strength of human dentin under diametral compression depends on the dentin tubular orientation in sample and varies from 40 MPa to 60 MPa [7]. However, no information on the type of deformation and fracture behavior was presented in this work.

Deformation behavior of dentin varies from brittle to elastic–plastic in dependence on both shape of sample and deformation scheme. It was shown that the type of its mechanical response is ruled by the proportion between tensile and compression stresses into the sample. Elastic–plastic behavior is observed when compressive stress is dominant, whereas brittle behavior is induced by predomination of tensile stress [8]. Indeed, dentin behaves like a brittle substance under tensile test [9–12]. Unfortunately, such experiments need high skills and, hence, cannot be used for routine examination of mechanical properties of human dentin. On the contrary, diametral compression testing allows estimating the tensile strength of dentin by the more easily way.

The organic matrix gives the dominant contribution into the plastic response of human dentin at room temperature (300 K) on microscopic level. This contribution could be diminished if mechanical testing is carried out at liquid nitrogen temperature (77 K), when an organic substance lost ability to the plastic response on applied stress. Also, it should be noted that such experiment is the simplest procedure in the low temperature mechanical testing of materials. Besides, collagen fibers could inhibit the crack growth in dentin [13]. Diametral compression of human dentin at room and liquid nitrogen temperatures allows examining its fracture behavior when the plastic channel for accommodation of elastic energy due to organic matrix is excluded. Therefore, the aim of this work is comparison of deformation behavior of human dentin under diametral compression at room and liquid nitrogen temperatures. This finding gives an opportunity to verify the hypothesis that plasticity of dentin under tensile stress is exclusively provided by the collagen fibers.

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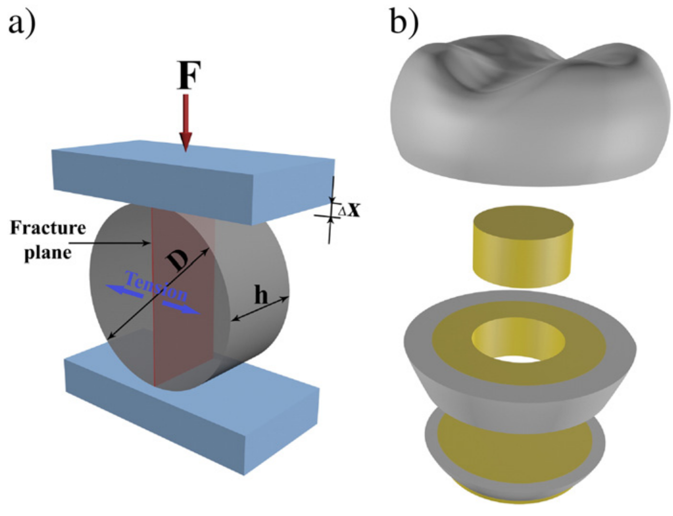

Fig. 1. The schemes: a — diametral compression testing; b — the sample preparation for diametral compression testing.

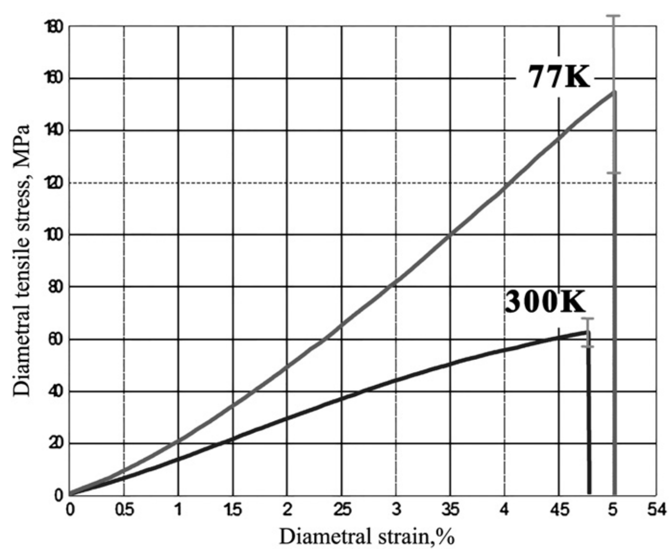

Fig. 2. Deformation curves of human dentin under diametral compression: red curve — 77 K; blue curve — 300 K (the green bars are the standard deviation for the diametral tensile strength).

2. Materials and methods

Fifty intact human molars and premolars without any visible damages, which were extracted from mature subjects according to the medical diagnosis and the Ethic Protocol of the Urals State Medical University at Ekaterinburg, have been used. All teeth were stored in 5% water solution of NaCl before the cut off. The samples for mechanical testing have been cut by the diamond tools with water irrigation from the crown of teeth according to Fig. 1b. Their shape was cylindrical or tablet with the diameter of 3 mm and 1.5 mm in height. Using scheme guarantees the uniform orientation of dentin tubulars in the samples, when they lay along the height of the tablet. Fifty samples were prepared; half of the samples were tested at room temperature, whereas the other half were tested in liquid nitrogen. All samples were washed in water jet and, further, they were dried in air.

Shimadzu AGX-50kN (Japan) was used for the mechanical testing in both temperatures [2]. Traverse rate was 0.1 mm/min for all tests. Foam plastic cavity was mounted on the lower punch, where liquid nitrogen has been poured at the low temperature test. The environment covers the sample during the test. Processing of the results and statistical analysis were carried out by Trapezium-X (Japan) that is the standard software of the Shimadzu facilities. The average and the standard deviation are used for presentation of the mechanical characteristics of dentin. Diametral tensile stress and diametral strain were calculated by $\sigma = \frac{2F}{\pi Dh}$ and $\varepsilon = \frac{\Delta x}{D}$, respectively (notations are given in Fig. 1a). The Young's modulus is calculated from the slope of linear part of the deformation curve. The diametral tensile strength is taken as the maximal stress on the deformation curve. The diametral elastic deformation of samples is estimated from the length of linear part of the deformation curve, whereas the proportional limit is taken as the maximal stress on the linear part of the deformation curve. The diametral plastic deformation is calculated from the length of nonlinear part of the deformation curve. The total deformation is the deformation of samples on the entire curve [14]. Surface of the samples was examined with a help of the MIM-8M light microscope (LOMO, USSR) under magnifications ×100 and ×500.

3. Results

The samples fail in the end of testing. Deformation curves at 77 K and 300 K, which are the most close to average curves for both groups, are presented in Fig. 2. The nitrogen curve can be approximated by a straight line, excepting the short non-linear part in its beginning. This non-linearity may be accepted as the artifact, caused by the deflection of the shape of sample from the ideal geometry, which is enhanced in the liquid nitrogen environment. The room temperature curve may be divided into two parts: linear (from zero to 3.6%) and nonlinear. The total deformation of samples, which includes elastic and plastic responses, is similar for both cases (~5%), while strength at 77 K is more than at 300 K. The mechanical characteristics of dentin samples are stored in Table 1.

Metallographic examination has shown that the shape of samples after testing at room temperature transforms from cylindrical to ellipsoidal, while at 77 K the shape does not change (Fig. 3). Crack trajectories lay along the line of compression of the sample (Fig. 3b and c). At 77 K the samples are always separated into two parts and the crack coasts have broken profile, as well (Fig. 3b). At room temperature, the samples are sometimes separated into three parts: the middle part, whose shape is closed to parallelepiped, and two back ones closed to a segment. Examination of the surface of samples tested at 300 K immediately before failure has shown that there is extensive cracking in the middle part of sample (Fig. 3c). It is important to note that despite the long cracks having similar sizes, not all of them become dangerous or its growth leads to failure. Cracks in room temperature samples develop along rectilinear trajectory, but their profile is rough on microscopic scale (Figs. 3c and 4). Every crack in room temperature samples consists of few parts including long and

Table 1
Mechanical properties of human dentin under diametral compression at 300 K and 77 K.

Environment	Young' modulus, GPa	Proportional limit, MPa	Diametral tensile strength, MPa	Diametral elastic deformation, %	Diametral plastic deformation, %	Total deformation, %
Air (300 K)	1.50 ± 0.09	59.6 ± 3.6	62.6 ± 5.2	3.6 ± 0.2	1.1 ± 0.3	4.7 ± 0.6
Nitrogen (77 K)	3.29 ± 0.41	155.5 ± 30.6	155.5 ± 30.6	5.0 ± 1.4	0.0 ± 0.0	5.0 ± 1.4