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Deformation behavior of human enamel and dentin–enamel junction under compression

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ABSTRACT

Deformation behavior under uniaxial compression of human enamel and dentin–enamel junction (DEJ) is considered in comparison with human dentin. This deformation scheme allows estimating the total response from all levels of the hierarchical composite material in contrast with the indentation, which are limited by the mesoscopic and microscopic scales. It was shown for the first time that dental enamel is the strength (up to 1850 MPa) hard tissue, which is able to consider some elastic (up to 8%) and plastic (up to 5%) deformation under compression. In so doing, it is almost undeformable substance under the creep condition. Mechanical properties of human enamel depend on the geometry of sample. Human dentin exhibits the similar deformation behavior under compression, but the values of its elasticity (up to 40%) and plasticity (up to 18%) are much more, while its strength (up to 800 MPa) is less in two times. Despite the difference in mechanical properties, human enamel is able to suppress the cracking alike dentin. Deformation behavior under the compression of the samples contained DEJ as the same to dentin. This feature allows a tooth to be elastic–plastic (as dentin) and wear resistible (as enamel), simultaneously.

1. Introduction

Dental enamel is the hardest tissue in a human body, which protects reliably a tooth from both mechanical loading and oral environment. Enamel possesses a complicated hierarchical structure, where at least three levels could be pointed. The first one is hydroxyapatite nanofibers (microscopic scale) that are bundled together to enamel rods (the second one or mesoscopic scale), whereas the third macroscopic one is an ordinate decussated structure of the rods [1–3]. There is lack of the experimental data on deformation behavior of human enamel under compression in the literature. According to the early works, dental enamel exhibits low ability to deformation prior the failure (~1%) at the ultimate compressive strength (UCS) $\sigma_c = 100–400$ MPa and Young's modulus $E = 10–80$ GPa (see Table 1) [4–6]. Such type of mechanical response is determined as brittle [7]. Resent finding obtained on a bovine enamel confirms that it behaves like a brittle substance, while its UCS is considerably higher and reaches ~700 MPa at Young's modulus 30 GPa [8]. The wide variation of the mechanical characteristics given in the literature may be caused by the fact that properties of enamel samples depend on the area of tooth, where they have been taken, or in other words, on the orientation of enamel rods in the sample [4,5]. Another possible cause for such behavior is the influence of geometry of enamel sample on its mechanical characteristics [9]. Also, it should be especially noted that some works have been carried out on a bovine enamel [8] and its mechanical properties can differ from a human enamel. This uncertainty is the obstacle for better understanding of physical mechanisms for stress accommodation in human enamel.

Human dentin, which is the hard basis of a tooth, exhibits the similar mechanical properties to enamel under compression: deformation prior the failure ~2%, UCS $\sigma_c = 250 \div 350$ MPa and Young's modulus $E = 7 \div 17$ GPa (see Table 2). Consequently, deformation behavior of dentin may be also characterized as brittle. However, it was recently shown that a human dentin can demonstrate the opposite deformation behavior under uniaxial compression. According to these findings, dentin is the high elastic (up to 40%) and plastic (up to 18%) at the high strength (UCS up to 800 MPa) material. At that its mechanical properties strongly depend on the geometry of sample [14]. Hence, it allows resuming that a human dentin can behave alike some filled polymers [15]. Besides, human dentin is able to consider elastic–plastic response under indentation, which suppresses the cracking near imprints [16]. Under point loading a human enamel also exhibits elastic and plastic deformation behavior, in spite of its difference in microhardness four times from a human dentin ($H_{Venamel} \sim 4$GPa, $H_{Vdentin} \sim 1$GPa) [8,16–19]. There is an unexpected result that brittle enamel under compression is able to consider elastic–plastic response under indentation. This difference in deformation behavior of enamel under compression and point loading can be caused by either intrinsic properties of this hard tissue on the macroscopic and mesoscopic levels or the features of deformation schemes applied. Unfortunately, experimental data on the

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Table 1
Compressive properties of enamel.

Authors	d/h	Rods orientation	UCS, MPa	Young's modulus, GPa
Stanford et al. (1958) [4]	Not recorded	Variable	277 ± 12.4	47.5
		Across	194 ± 15.2	30.3
		Along	134 ± 27.9	8.96
Stanford et al. (1960) [5]	Not recorded	Variable	261 ± 41.4	46.2 ± 4.8
		Across	250 ± 29.6	32.4 ± 4.1
		Along	94.5 ± 32.4	9.65 ± 3.45
		Along	126.9 ± 30.3	11.0 ± 2.1
Craig et al. (1961) [6]	1–0.25	Along	384 ± 85.3	84.1 ± 6.2
		Along	372 ± 56	77.9 ± 4.8
Ang et al. (2010) [8]	2.8	Side enamel	680	30

compression of human enamel in the literature is lacking but better understanding of the problem could be reached.

The macroscopic condition for the high cohesive strength of the joining of two materials is the similarity of their mechanical properties, whereas the microstructure of the boundary between them plays an important role in the physical mechanism of joining. It is well-known that a dentin–enamel junction (DEJ) in an intact tooth is mechanically stable and plays the important role in accommodation of mechanical stress [20–22]. However, it is unclear how a stable joining can be formed between brittle enamel and elastic–plastic dentin? Taking into account elastic–plastic response of human enamel under point loading, it is reasonably supposed that human enamel is able to consider elastic–plastic behavior under compression, too. Therefore, the aim of this work was the experimental study of human enamel and DEJ under compression for the search for the characteristics of their macroscopic responses on the external stress.

2. Experimental

Thirty human molars, which did not contain any visible damages/pathologies, were used in this research. The teeth were extracted from the patients of 20–40 years old according to the medical diagnosis and the ethic protocol of the Ural State Medical Academy at Yekaterinburg, Russia. Samples of enamel and DEJ have been cut from the back side of the crown part of tooth using the diamond saw with water irrigation as it is shown in Fig. 1. Such scheme for cutting allows reaching the maximal size of enamel samples for mechanical testing. The dentin samples have been prepared from the crown dentin according to technique described in ref. [14]. Samples were distributed on the six groups: the four groups consist of the enamel samples having different sizes (d/h ratio, see Figs. 2 and 3a), the fifth group is the samples which contained DEJ (Fig. 3b) and the sixth one consists of the sandwiches "dentin-on-enamel" (Fig. 3c), where the enamel sample lays on the dentin one without any bonding (see Table 3). Only three d/h ratios have been used for the study of the shape effect in human enamel. It is considerably less than ten groups with different d/h ratios used for the study of the shape effect in human dentin [14]. The small volume of enamel in a tooth in comparison with dentin is the main cause for such decision, why d/h ratios of the enamel samples lay in the frames of 2.2–6.4. The difference in d/h ratios between the groups was connected with the fact that the back size of samples from different groups differs in 0.5 mm (Table 3). All samples were mechanically polished by abrasive papers having different sizes of grit down to 1000. After that they were washed in water jet during 15 min and stored in water until the test. Before the testing, the samples were dried in air during 1 h. The working surfaces of each sample were examined by optical microscopes in transmission and reflected light at magnifications ×20 and ×500. It allows detecting cracks both on the surface and in the volume of samples. Metallographic examination of the samples prior compression tests has shown that: (1) there are no cracks in the samples; and (2) the enamel rods have the similar orientation in all enamel samples — they are inclined to the compression axis on the angle ~45° (see Fig. 4). It must be noted that the mentioned above orientation of enamel rods cannot be considered as an exact characteristic of the samples, because all enamel rods in such macroscopic volumes never can be oriented uniformly. Therefore, this angle points to the prevailing orientation of the enamel rods in the samples only. The compression tests were carried out on Shimadzu AGX-50kN testing machine at the room temperature. The Trapezium™ software was used for the processing of experimental data. The groups No 1–3, 5, and 6 were compressed at the traverse rate 0.1 mm/min, whereas the group No 4 was tested under the creep condition (5 h under 200–450 MPa). The size of samples was measured by the optical instrumental microscope UIM-21 (precision of measurement 1 μm) prior and after each test.

3. Results

Compression testing is stopped as soon as the drop of stress happens or the jog appears on the deformation curve. All samples of group 1 demonstrate the similar deformation behavior and, therefore, their typical compression curve is given in Fig. 5 (the curve d/h = 4.3). The appearance of the jog is caused by initiation and growth of many cracks, which are clearly visible on the compression surface of sample (see Fig. 6). It is important to note that these cracks do not cause the failure of samples immediately, but sometimes small pieces of enamel are separated. The total deformation of the samples from this group is ~11% at the ultimate stress ~550 MPa that may be accepted as its UCS.

Table 2
Compressive properties of dentin.

Authors	UCS, MPa	Young's modulus, GPa
Black (1895) [10]	256 ± 30.3	–
Peyton et al. (1952) [11]	250 ± 22.6	11.5 ± 1.2
Stanford et al. (1958) [4]	348 ± 24.5	11.4 ± 1.7
Craig & Peyton (1958) [12]	297 ± 24.8	16.6 ± 1.8
Stanford et al. (1960) [5]	Crown 282.6	Crown 12.5
	Root 233.5	Root 7.75
Watts et al. (1987) [13]	270.9 ± 7.0	13.94 ± 1.5

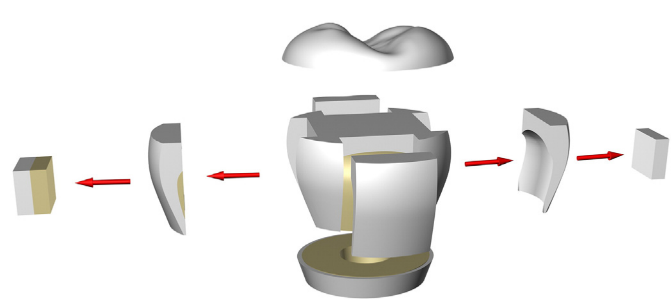

Fig. 1. Preparation of the enamel and DEJ samples for compression tests.

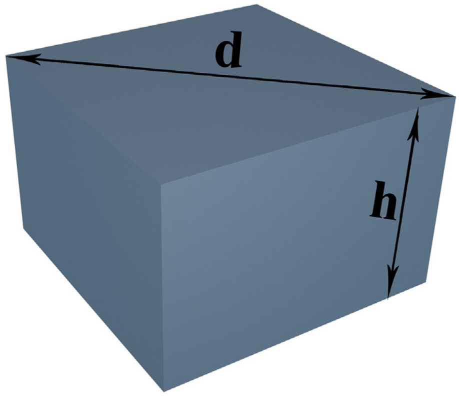

Fig. 2. Geometry of dentin samples for compression test.

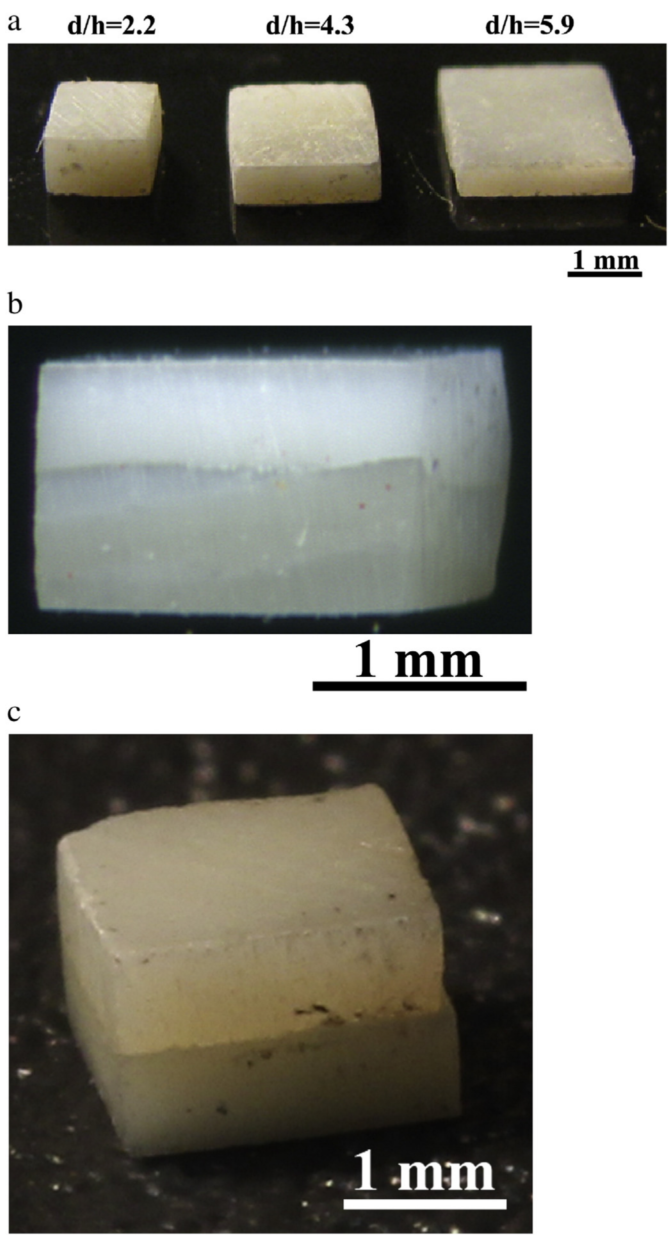

Fig. 3. The enamel samples: a — the samples with different d/h ratio, groups 1–4; b — the samples contained DEJ, group 5; c — the sandwich "enamel-on-dentin", group 6.

Table 3
Characteristics of the sets of samples.

Set	Tissue	Quantity of samples	Size			
			a, mm	b, mm	h, mm	d/h
1	Enamel	30	2	2	0.65	4.3
2	Enamel	5	1.45	1.45	0.95	2.2
3	Enamel	5	2.5	2.5	0.55	6.4
4	Enamel	5	2	2	0.65	4.3
5	DEJ	5	2	2	1.3	2.2
6	Enamel	5	2	2	0.65	2.2
	Dentin	5	2	2	0.65	

The measurement of the samples size prior and after the test has shown that irreversible/plastic deformation of enamel reaches ~3%, while reversible/elastic deformation, which was determined as the difference between the total deformation and the irreversible deformation of sample, is ~8%. Young's modulus is estimated as ~7 GPa from the slope of compression curve on the elastic region despite that it is nonlinear. Mechanical properties of these samples (the average values with their standard deviations) are stored in Table 4. The samples having other d/h ratios were tested, too (see Fig. 3a). The typical compression curves of the samples from groups 2 (d/h = 2.2) and 3 (d/h = 6.4) are presented in Fig. 5. A lot of cracks appear in all tested samples, which also never fail. The total deformation of the samples with d/h = 6.4 is the same to the previous case, whereas its UCS is ~1850 MPa. On the contrary, samples of group 2 give the total deformation ~4% and the UCS ~250 MPa. Young's modules are ~25 GPa and ~9 GPa, respectively. The proportion between elastic and plastic contributions in the total deformation is close to 1:1 for both groups. The findings are given in Table 4, too.

Group 4 was tested under the creep condition. The samples hold the applied load without failure during 5 h when stress is ≤450 MPa (Fig. 7a). No cracks were observed on the compression surfaces of samples after the test. However, they are immediately separated if applied stress is close to the UCS of the sample (≥500 MPa). It was shown that the magnitude of deformation under constant loading (ε_t) increases slowly with applied stress growing, but it never exceeds of 1% (Fig. 7b).

The typical deformation curves for sample contained DEJ (group 5) and sandwich "enamel-on-dentin" (group 6) are given in Fig. 8. The curve of enamel sample from group 2 (d/h = 2.2) and the curve of dentin sample having d/h = 2.2 [14] are also presented here for comparison. Deformation behavior of DEJ and dentin samples is almost similar: elastic deformation ~7%, plasticity ~4%, UCS ~400 MPa and Young's modulus ~5GPa. The typical curve of the sandwich is situated between enamel and dentin curves: elastic deformation ~5%, plasticity ~2%, UCS ~400 MPa and Young's modulus ~7 GPa. Contribution in the total plastic deformation of enamel and dentin blocks of the sandwich is obtained from the differences of their sizes prior and after testing. It was calculated that the irreversible deformation of dentin block is about 2%, while the size of enamel block does not change. No DEJ samples and sandwiches "dentin-on-enamel" are separated under compression. In DEJ samples, a lot of cracks appear in the enamel part, but they never pass via the DEJ into the dentin part. Estimation has shown that the quantity of cracks in the enamel block of the sandwich is much more than in the dentin one (Fig. 9). Mechanical properties of these samples are collected in Table 5.

4. Discussion

Compression tests have shown that human enamel possesses both elasticity (up to 8%) and plasticity (up to 5%) and its mechanical characteristics depend on the d/h ratio of sample. Human dentin is also prone to the similar behavior, but its values of elastic (up to 40%) and plastic (up to 18%) deformation are more than for enamel. However, the strength of dentin (up to 800 MPa) is less than for the

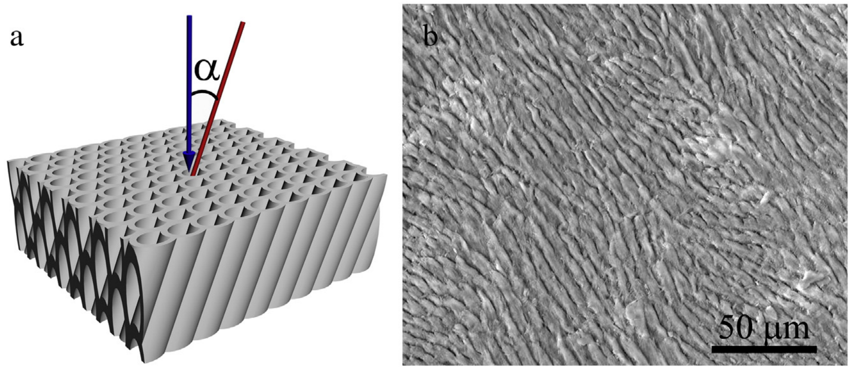

Fig. 4. Orientation of the enamel rods in the samples: a — angle between the compression axis and the enamel rods in the sample; b — back surface of the sample.

enamel samples with d/h ratio = 6.4 (~1850 MPa), while their strengths are comparable for the samples, whose d/h <6 [14]. The shape effect in enamel has some common features with the shape effect in dentin. For example, the samples having d/h ratio 2.2 and 4.3 display the same tendency of changing their mechanical properties. Young's modulus decreases when d/h ratio grows, while UCS and elastic and plastic contributions increase (see Table 3). The enamel samples with d/h = 6.4 do not conform to this trend: their elasticity does not grow up and Young's modulus increases considerably. Equivalent to dentin, detail study of the shape effect in enamel (on the ten d/h ratios) did not fulfill because there is a small volume of enamel in a tooth.

The difference of some mechanical characteristics of human enamel in comparison with ref. [4–6] may be explained by the different geometries of samples (see Table 1). The analysis of deformation behavior of the bovine enamel samples with the similar d/h ratio (2.8) has shown that they also demonstrate elastic and plastic deformations under compression, but their magnitudes are lower than for the human enamel, whereas its UCS and Young's modulus are higher (see Table 1, [8]). Besides, the samples of bovine enamel do not separate under compression as it takes place in human enamel. On the other hand, the distinction between the literature data and our findings may be also connected with the different distributions of enamel rods in the samples. The exact orientation of the enamel rods can be realized in the tiny samples only, whose size is comparable with the size of the few enamel rods. In this case, the macroscopic level — an ordinate decussated structure of the rods is practically excluded from the factors' rule by the deformation behavior of material. Therefore, determination of the relationship between orientation of the rods and deformation behavior of human enamel, when all three structural levels contribute into the accommodation of external stress, becomes impossible. Point loading gives an opportunity to describe the deformation behavior of enamel on the micro- and the meso-scopic scales. Indentation experiments have shown that Young's modulus along the enamel rods is more than Young's modulus measured perpendicularly to the enamel rods' direction [3,23]. However, these results contradict with the data on compression of enamel in ref. [4,5], where UCS and Young's modulus are less in the direction along the enamel rods than in the normal direction. This fact points to the importance of an ordinate decussated structure for the accommodation of stress in dental enamel. Hence, microindentation cannot describe correctly its deformation behavior and this information should be added by the macroscopic mechanical tests.

The samples of enamel from group 3 (d/h = 6.4) exhibit the highest value of UCS (~1850 MPa) in comparison with the dentin samples having the maximal UCS (~800 MPa at d/h = 9.8), whereas this difference for the samples having another geometry is insufficient. In both cases, they are plane samples, where under compression macroscopic wedge stress is minimal and, hence, crack growth should be suppressed here. It is well-known that there are two channels for the accommodation of elastic energy in solids — deformation and

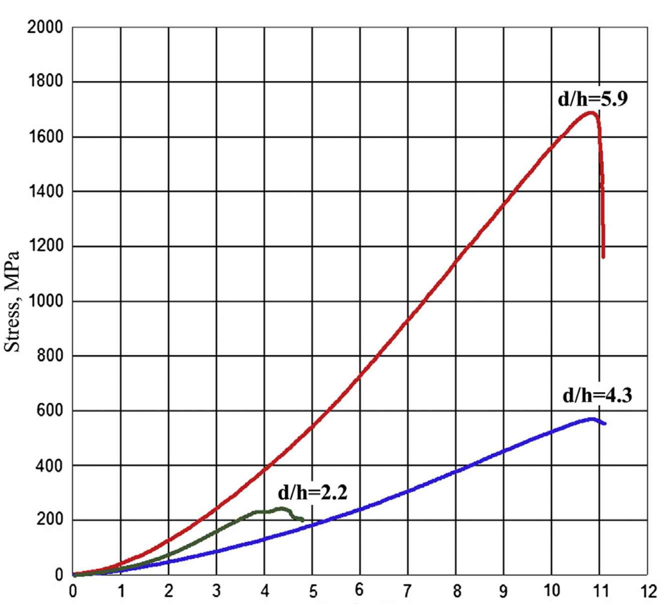

Fig. 5. Stress–strain curves of the enamel samples with different d/h ratios (Groups 1–3).

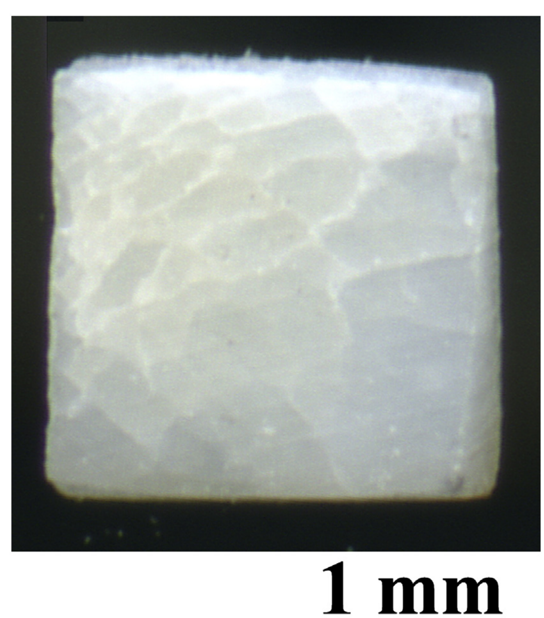

Fig. 6. The compression surface of the enamel sample after compression (Group 1).

Table 4
Mechanical properties of human enamel samples with different d/h ratio under compression.

d/h	Set	Young's modulus, GPa	UCS, MPa	Elastic deformation, %	Plastic deformation, %	Total deformation, %
2.2	2	8.79 ± 1.68	241 ± 56	1.8 ± 0.8	2.4 ± 0.3	4.2 ± 0.8
4.3	1	6.80 ± 1.38	538 ± 87	7.7 ± 2.0	3.5 ± 1.9	11.2 ± 1.0
6.4	3	24.91 ± 2.08	1850 ± 120	4.6 ± 1.8	5.3 ± 2.4	9.9 ± 0.6

cracking. In the case, when one of them has been suppressed, all external stresses should be relaxed by means of elastic–plastic deformation. As a result, UCS of the plane samples cut from the tooth hard tissues should be the most high. Dentin has the channel structure on the mesoscopic level. Therefore, its porosity or the density of the microstress concentrators is considerably higher compared with enamel, where the same defects are absent. As a result, the probability of crack nucleation in dentin is more than in enamel. Due to the high content of the calcium hydroxyapatite in human enamel (~97%) and homogeneous microstructure on the mesoscopic level, the UCS of the samples of group 3 is close to this parameter for the hydroxyapatite. Considerable growth of Young's modulus of enamel samples from group 3 also supports this conclusion, insomuch as this parameter for the calcium hydroxyapatite reaches 70 MPa, while an influence of hierarchic structure on the strength of the plane samples drops. Solid state mechanics consider Young's modulus as the elastic constant

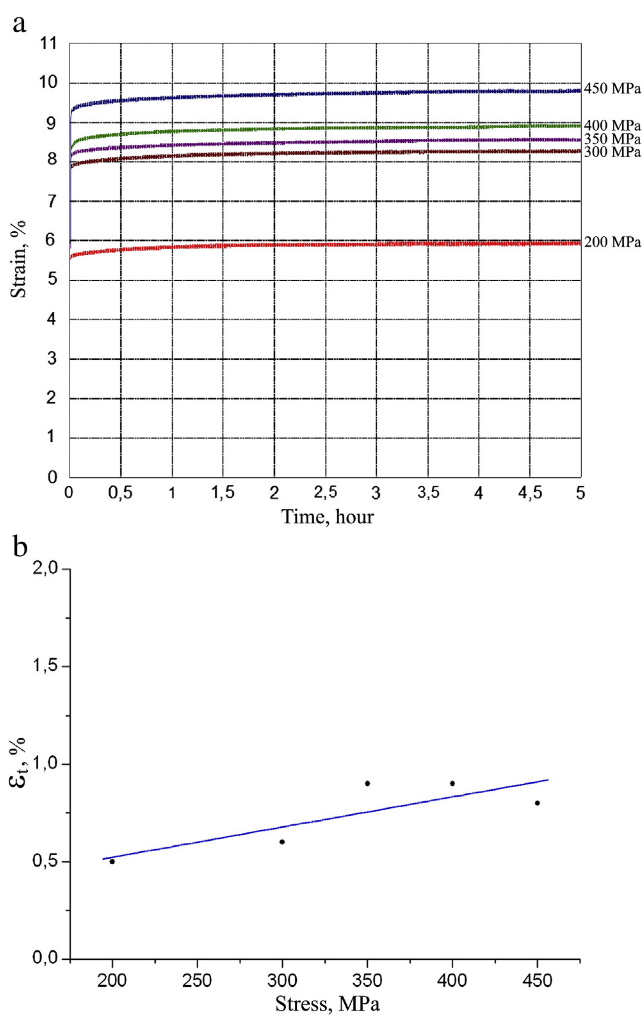

Fig. 7. Creep of the enamel samples: a — strain–time deformation curves for different loads; b—dependence between ε_t and holding stress, ε_t — strain under holding of constant stress.

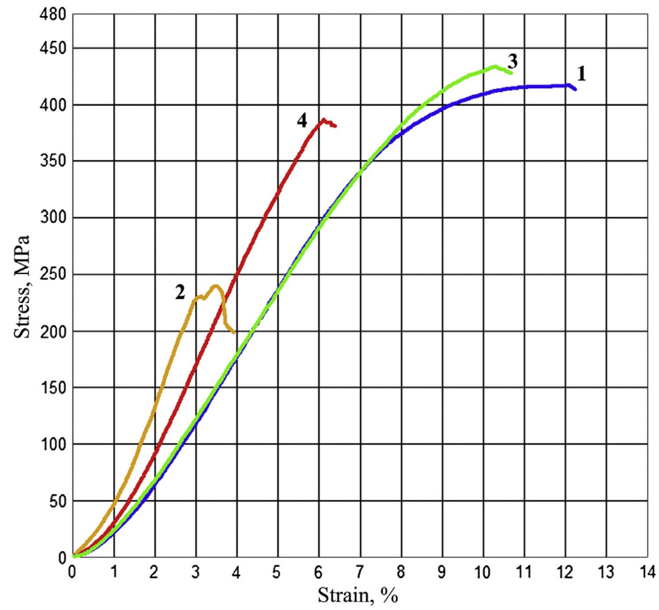

Fig. 8. Stress–strain curves of the dentin–enamel samples: curve 1 — dentin; 2 — enamel; 3 — DEJ; 4 — sandwich "enamel-on-dentin".

characterized deformation behavior of material [7]. Experiments have shown that under compression brittle solids, which are not able to considerable deformation, are subject to this rule only. Elastic–plastic solids, where deformation is the dominant channel for accommodation of elastic energy, do not obey it and the slope of deformation curve on the elastic region/Young's modulus can vary. In the high samples (groups 1 and 2), where the level of wedge stress is higher than in the plane ones, a porosity of a tooth hard tissue on the mesoscopic level does not play a decision role. These arguments allow understanding better why the shape effect possesses distinct tendencies for the plane samples of dentin and enamel.

It may be stated that on the macroscopic level human enamel behaves alike human dentin and some filled polymers. However, the small values of elastic and plastic responses and the high strength point to that deformation behavior of enamel is sooner close to some hard rock materials, for example, basalt, granite, and quartzite, than to a rubber [24]. The enamel samples with d/h = 6.4 (the plane ones) are more inclined to the hard rock materials, whereas the samples having d/h = 4.3 are similar to filled polymers. This feature of enamel may be explained by its high mineral content ~97% in comparison with dentin ~70% [20]. This conclusion is also supported by the fact that enamel is almost an undeformable substance under creep conditions.

Fracture behavior of human enamel is similar to dentin, too. Many cracks appear in the sample under the compression test, but it does not lead to the failure immediately. The total amount of cracks in the enamel sample is much more than in the dentin one having the same size (Fig. 6). The enamel samples always separate under the second loading, whereas the dentin ones can be compressed few times without failure [14]. It may be concluded that there are effective mechanisms for crack growth termination in both enamel and dentin. These mechanisms were considered in ref. [25,26] for the fatigue condition. In the tooth hard tissues, the main crack develops due to joining with the satellite cracks ahead its tip. The same picture was observed in thin dentin and enamel samples under tension in transmission light and transmission electron beam. However, there is clearly visible plastic zone, where the pore-like/satellite cracks nucleate and advance, ahead the main crack as it takes place in thin metallic foils for transmission electron microscope [27–29]. Consequently, no qualitative difference in the deformation and fracture behavior under compression of these tooth hard tissues is revealed, despite that human dentin is the more deformable substance than enamel. Elasticity and plasticity are the

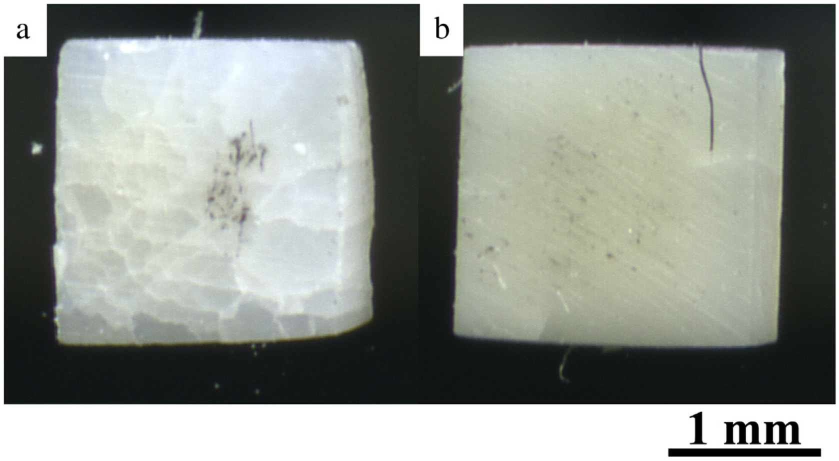

Fig. 9. The compressive surface of sandwiches “enamel-on-dentin”: a — enamel block after compression; b — dentin block after compression.

concurrent channel for the accommodation of elastic energy to the crack growth. As a result, dentin must suppress cracking more effectively than enamel. Indeed, the findings obtained in the DEJ samples agree with this statement. The majority of cracks in the samples nucleate in the enamel parts and do not propagate via DEJ into the dentin part. This effect has been discussed in the literature [30–32]. On the basis of data presented above, the similarity in mechanical properties of human dentin and enamel may be considered as the main cause of strength of DEJ in a human tooth.

The comparison of deformation behavior of the DEJ samples (group 5) with the sandwiches “dentin-on-enamel” (group 6) allows estimating the contribution of DEJ into the stress accommodation in a tooth. The DEJ sample behaves like dentin, while the deformation curve of “dentin-on-enamel” sample is situated between the dentin and enamel ones. Hence, the presence of DEJ between enamel and dentin in a tooth makes this natural composite wear-resistible as enamel and highly deformable as dentin. This mechanism of stress accommodation is caused by the microstructure of DEJ and needs in additional structural study.

5. Conclusion

It was shown for the first time that human enamel exhibits elastic–plastic behavior under compression test. It is the strength substance (up to 1850 MPa), which is able to consider some elastic (up to 8%) and plastic (up to 5%) deformation, in so doing its mechanical characteristics depend on the shape of sample (d/h ratio). Such macroscopic deformation scheme as the uniaxial compression allows estimating the total response of a multilevel composite material in contrast with point loading (micro- and nano-indentation), which are limited by the mesoscopic and microscopic scales. Human dentin demonstrates the similar deformation behavior including the shape effect, but the values of its elasticity and plasticity are much more, while its strength is less in two times. Despite this difference, the compression tests have shown that DEJ is mechanically stable and plays the important role in deformation behavior of a tooth.

Table 5
Mechanical properties of DEJ samples under compression.

Set	E, GPa	UCS, MPa	Elastic deformation, %	Plastic deformation, %	Total deformation, %
–	5.46 ± 0.35	406 ± 25	7.0 ± 0.7	4.7 ± 1.5	11.7 ± 2.0
2	8.79 ± 1.68	241 ± 56	1.8 ± 0.8	2.4 ± 0.3	4.2 ± 0.8
5	5.31 ± 0.48	428 ± 12	6.6 ± 2.0	3.6 ± 1.0	9.9 ± 1.7
6	6.87 ± 0.90	379 ± 41	–	0.0 ± 0.5	6.6 ± 0.9
			–	1.6 ± 0.5	

Acknowledgments

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