Common Features of Deformation Behavior Between Human Tooth Enamel and Rocks

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Article history	Abstract	
Received August 28, 2023 Accepted September 04, 2023 Available online September 30, 2023	The contribution of bioorganic components in the deformation behavior of a rock-like biocomposite human tooth enamel is discussed. Uniaxial compression testing and Brazilian testing (diametral compression) in liquid nitrogen (77K) and in air at room temperature were carried out on the samples cut from human tooth enamel. It was compared with deformation behavior of some rocks (granite, serpentinite, and jasper) and plasma-sprayed Al ₂ O ₃ under compression and Brazilian testing in air at room temperature. It was shown that enamel and the rocks exhibit the viscoelastic-like deformation behavior under compression, whereas their macroscopic response becomes brittle under tensile stress. Fracture surface morphology was attested as brittle in all model materials, although cracks in them all advance by the viscoelastic-like manner as a crack in a ductile metal. The contribution of viscoelastic bioorganic component in deformation behavior of enamel is detected at room temperature only because bioorganic component leaves the viscoelasticity at low temperatures. However, this contribution does not lead to changing the character of deformation behavior of the rock-like biocomposite in comparison with these rocks.	

Keywords: Human tooth enamel; Rocks; Compression; Brazilian testing; Deformation behavior

1. INTRODUCTION

Human tooth enamel is the most mineralized tissue in a human body composed of 95 weight % of inorganic component, namely, calcium hydroxyapatite, and 5 weight % of bioorganic compounds [1]. Therefore, sometimes it is called a mineral of biological genesis or a biomineral [2]. Tooth enamel possesses a complicated hierarchically organized structure, which is usually absent in rocks and minerals [3]. The basic morphological feature of human tooth enamel is the fibril-like hexagonal calcium hydroxyapatite crystallites joining into the enamel rods. The rods appear in vicinity of dentinoenamel junction and propagate to the occlusal surface of a tooth. Enamel rods are parallel oriented one to another and directed perpendicularly to both dentinoenamel junction and occlusal surface. The bioorganic phases are situated in intercrystallite and interrod space of enamel [3,4]. Despite small amount of bioorganic component, it can play the important role in the deformation behavior of tooth enamel [5,6]. Indeed, crack growth in enamel occurs namely in an interrod space [7].

According to main chemical content, tooth enamel and rock are inorganic materials, which should behave in brittle manner under mechanical loading [8], while their deformation behavior should not dramatically depend on a structure of a solid. Comparison of deformation behavior of human tooth enamel and some rock materials under the same conditions allows better understanding of the contribution of viscous bioorganic component to deformation behavior of this biomineral. Solving this task has certain interest in materials science because some mechanical properties of human tooth enamel could sound as a contradiction with an empirical knowledge on rock materials [9].

Combination of the uniaxial compression and the diametral compression (Brazilian testing) that could be carried out on the samples with same shape and size give an opportunity to describe the deformation behavior of a brittle material under both compression and tensile loadings, including crack growth on the surface of the samples. Mechanical

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testing at low temperature should cancel an influence of viscous bioorganic phases on the deformation behavior of tooth enamel because an organic compound becomes brittle under these conditions [10]. Hence, examination of the deformation behavior of human enamel in liquid nitrogen (77 K) allows estimating the contribution of viscoelasticity of the bioorganic phase to its deformation behavior. Similar study was carried out in the human dentin [11]. The aim of this work is the comparison of deformation behavior of human tooth enamel under compression and tension at room temperature and in the liquid nitrogen at 77 K with deformation behavior of some rocks. The findings obtained may be helpful for better understanding of the stress accommodation mechanisms in human tooth enamel and some rocks.

2. MATERIALS AND METHODS

Over two dozen of human teeth without visible damages were extracted from mature subjects of 25-40 years old according to the Ethic Protocol of Urals State Medical University at Ekaterinburg in Russia. Forty enamel samples were cut off from these teeth by means of the diamond saw under water irrigation by the technique used in Ref. [11]. All samples consisted of the intermediate enamel, where enamel rods were inhomogeneously distributed in the volume of the sample. Sample with uniform orientations of the enamel rods cannot be prepared due to the feature of human enamel morphology. The working surfaces of the samples were abraded using the abrasive papers and polishing pastes with different grain sizes for removing the damaged layer, which appeared under cutting off procedure. Samples for uniaxial compression (20 pieces) had the shape of parallelepiped with the size of 2×2×0.7 mm³. Samples for diametral compression / Brazilian testing (20 pieces) possessed the cylindrical shape with the diameter of 2.5 mm and the height of 1.25 mm. The samples were distributed into two sets (10 pieces per each) for every deformation scheme. The first set was tested at room temperature (300 K), while the second one was tested in the liquid nitrogen (77 K). Granite, serpentinite, and jasper from deposits of the Ural region (Russia) were chosen as the model rocks for comparison of deformation behavior with human enamel. Plasma-sprayed alumina was also used as the model inorganic material for comparison. Samples having a tablet shape (6 mm in diameter and 3 mm in thickness) were cut from massive workpieces by the drilling of hollow diamond drill in water. Two sets of samples of every model material per ten pieces in every set were tested under compression and Brazilian testing at room temperature. The curve of the sample that is close to behavior of all samples from the set is chosen for

presentation as the deformation curve of model material in figures in the next chapter.

Two loading schemes, namely uniaxial compression and diametral compression (Brazilian testing), were used for mechanical testing in air at 300 K and in liquid nitrogen (77 K). Shimadzu[™] Autograph AGX-50kN testing machine was taken for the mechanical tests (traverse rate was 0.1 mm/min). The Trapezium[™] package (the software of ShimadzuTM) was applied for processing of the experimental data including the statistical analysis. Elastic modulus was calculated from the slope of the rectilinear part of deformation curve. The total deformation was obtained using the testing machine for both schemes of deformation. Maximal stress prior to the crack appearance in the sample under testing was accepted as the ultimate strength. Scanning electron microscope (SEM) JSM 6390LV was used for the examination of the fracture surfaces of samples. Working surfaces of the samples before and after testing were examined with the help of a light metallographic microscope.

3. RESULTS

The deformation curves of the model materials tested under different conditions are shown in Figs. 1–3, while their mechanical properties are collected in Table 1. Fracture surfaces of the tooth enamel, rocks and alumina after Brazilian testing are presented in Figs. 4–6, while cracks on their working surfaces are given in Figs. 7,8.

The deformation curves of tooth enamel under uniaxial compression in liquid nitrogen (77 K) and in air at room temperature are given in Fig. 1a, while the ones under Brazilian testing are shown in Fig. 1b. A straight line could approximate the course of the deformation curve of enamel under testing in liquid nitrogen, while the difference in maximal deformation reached one order (~8% and ~1% for uniaxial compression and Brazilian testing, respectively). Testing of tooth enamel in air has shown that a straight line, which, however, laid lower than its curve for liquid nitrogen, could also approximate the deformation curve for Brazilian testing. The maximum tensile stress of enamel at 77 K is higher than at 300 K (48 MPa and 40 MPa, respectively), while the maximal strain is vice versa (1.1% and 1.3%, respectively). The shape of deformation curves and maximum strain allow estimating deformation behavior of tooth enamel under Brazilian testing at both temperatures as brittle. The shape of the compression curve of enamel at 300 K could be divided in two parts (0-5% and 5.5-10%, respectively), both of which is approximated by a straight line. It is visible that the angle of inclination of the curve for the second part is higher than for the first one. The maximum compression stress of enamel at 77 K is higher than at room temperature



Fig. 1. Deformation curves of human tooth enamel in air at 300 K and in liquid nitrogen (77 K): a – under uniaxial compression; b – under Brazilian testing (diametral compression).



Fig. 2. Deformation curves of some rocks in air at 300 K: a – under uniaxial compression; b – under Brazilian testing (diametral compression).



Fig. 3. Deformation curves of plasma sprayed Al_2O_3 in air at 300 K: a – under uniaxial compression; b – under Brazilian testing (diametral compression).

Material	Elastic moduli <i>E</i> , GPa	Ultimate Strength σ _B , MPa	Deformation δ, %
	Unia	xial compression	
	in liqu	id nitrogen (77 K)	
Human tooth enamel	8.8 ± 0.8	580 ± 40	8.2 ± 0.5
	in	n air at 300 K	
Human tooth enamel	8.1 ± 0.3	520 ± 20	11.0 ± 0.2
Granite	6.8 ± 0.4	550 ± 30	9.5 ± 1.4
Serpentinite	6.1 ± 0.4	400 ± 30	6.2 ± 0.3
Jasper	7.5 ± 0.3	460 ± 30	6.8 ± 0.2
Plasma-sprayed Al ₂ O ₃	7.5 ± 0.5	420 ± 50	7.4 ± 0.2
	Brazilian testin	g (diametral compression)	
	in liqu	id nitrogen (77 K)	
Human tooth enamel	4.5 ± 0.9	48.5 ± 4.4	1.1 ± 0.1
	ir	1 air at 300 K	
Human tooth enamel	3.3 ± 0.3	39.9 ± 4.7	1.3 ± 0.1
Granite	1.5 ± 0.2	14.7 ± 3.8	1.1 ± 0.2
Serpentinite	1.5 ± 0.1	13.5 ± 1.4	1.0 ± 0.1
Jasper	3.0 ± 0.3	28.1 ± 1.5	1.2 ± 0.1
Plasma-sprayed Al ₂ O ₃	2.0 ± 0.2	30.0 ± 4.0	1.4 ± 0.2

Table 1. Mechanical properties of human tooth enamel and model rocks under uniaxial compression and Brazilian testing (diametral compression) in liquid nitrogen (77 K) and in air at 300 K.

(580 MPa and 520 MPa, respectively), while maximum compression deformation is lower (8% and 11%, respectively). The type of deformation behavior of tooth enamel under uniaxial compression cannot be attested as brittle due to the high value of compression. However, it should be noted that its behavior at 77 K is not like its behavior at 300 K.

A straight line could also approximate the course of compression curves of the model rocks at room temperature (Fig. 2a). The angles of their inclination are close to each other that is just expected, because all of them were metamorphic genesis. The maximum compression stress depends on the type of rock and varies from 400 MPa (serpentinite) to 550 MPa (granite). Maximum compression before the fragmentation/starting the failure of the sample laid in the limits of 5–10% depending on the type of rock that is very high for a brittle solid. Therefore, deformation behavior of these rocks under compression cannot be estimated as brittle despite a rectilinear course of deformation curves. A straight line characterizes the course of deformation curves of the model rocks under Brazilian testing when deformation does not exceed 1% at the ultimate stress of 150MPa (Fig. 2b). The inflection of a curve occurs under deformations that are higher than 1% and reaches 1.2-1.6% at 25-28 MPa. After that, however, a straight line continues to approximate the course of deformation curve. Such behavior should be attested as brittle despite the feature described above. The deformation behavior of the plasma-sprayed alumina under uniaxial compression at room temperature is like the rock materials, when the compression curve looks like a straight line (Fig. 3a). Maximum compression of 8–9% at 350 MPa does not allow calling this behavior as brittle as well. The brittle response of this ceramics happens under Brazilian testing: maximum deformation is about 1.3% at 30 MPa. At that, the inflection of the deformation curve also takes place when deformation exceeds 1% (Fig. 3b).

Cracked samples after Brazilian testing, except tooth enamel tested at 77K and jasper, were separated apart before SEM study of their fracture surfaces. Analysis of fracture surfaces morphology of the model materials under diametral compression confirms that there is brittle fracture in all examined samples (see Figs. 4-6). Typical SEM images of fracture surfaces of tooth enamel tested at 77 K and 300 K are given in Fig. 4 and Fig. 5, respectively. Comparison of fracture surfaces of the enamel samples and literature sources [1,5] has shown that the brittle transcrystalline fracture is fracture mode of human tooth enamel under these experimental conditions. No specific morphological features on both macroscopic and microscopic scales were found in the fracture surface of enamel tested in liquid nitrogen compared to the testing at room temperature. It is somewhat surprising because the enamel samples in liquid nitrogen always failed under loading, while crack growth was reliably stopped under Brazilian testing at 300K.



Fig. 4. Fracture surface (SEM) of human tooth enamel under Brazilian testing (diametral compression) in liquid nitrogen (77 K): a – low magnification; b – high magnification.



Fig. 5. Fracture surface (SEM) of human tooth enamel under Brazilian testing (diametral compression) in air at 300 K: a - low magnification; b - high magnification.



Fig. 6. Fracture surface (SEM) of some rocks under Brazilian testing (diametral compression) in air at 300 K: a - granite; b - serpentinite; c - jasper; $d - plasma-sprayed Al_2O_3$.

The fracture surface morphology of both granite (Fig. 6a) and serpentinite (Fig. 6b) was determined as the brittle transcrystalline fracture (see Ref. [12]). Despite this circumstance, samples of these rocks never separated apart under Brazilian testing. On the contrary, the cause why jasper samples always failed under diametral compression is grain boundary brittleness that occurs in this mineral [9]. Indeed, some portion of the brittle intercrystalline fracture is clearly observed in its fracture surface (Fig. 6c). Plasma-sprayed alumina behaves similarly to granite and serpentinite samples under the same conditions in Brazilian testing. Its fracture surface morphology is also estimated as the brittle transcrystalline fracture, but the scale of river patterns in alumina is considerably smaller than in these rocks (Fig. 6d).

As it was mentioned above, the dangerous crack, whose growth leads to failure of the sample under Brazilian testing at room temperature, could be observed in tooth enamel (Fig. 7), granite (Fig. 8a), serpentinite (Fig. 8b), and plasma-sprayed alumina (Fig. 8c). The trajectory of dangerous crack is determined by the geometry of applied stress in the loading scheme, namely diametral compression, when it moves approximately along the axis of loading, while tensile stress is directed perpendicularly to the crack growth direction [13]. The dangerous crack in the model materials possesses a broken profile and consists of coalesced and almost coalesced pore-like cracks. There are few short satellite cracks ahead every dangerous crack (Fig. 7b). Sometimes, cracks in the model materials have a sharp tip, as it should be in a brittle solid, while considerable width of cracks points to a significant contribution



Fig. 7. Main crack with satellite cracks ahead on the working surface of human tooth enamel under Brazilian testing (diametral compression) in air at 300 K (light microscope).



Fig. 8. Cracks on the working surface of some rock materials under diametral compression (Brazilian testing) in air at 300 K (optical microscope): a - granite; b - serpentinite; c - plasma-sprayed Al₂O₃.

of viscosity to the deformation behavior of the model materials (Fig. 7a, Figs. 8b,c). Besides, the picture of cracking in considered biomineral, rocks and ceramics looks similar to crack growth in plastic metals, namely in neck region of bulk samples [14] and in thin foils under in situ tension inside TEM column [15,16]. Hence, it may be concluded that cracks in the model materials, which exhibit the brittle deformation behavior under diametral compression at room temperature, grow in the viscous manner.

4. DISCUSSION

Analysis of the findings has shown that deformation behavior of the model materials is similar to each other and, hence, the response of the model material does not depend on its genesis (biological, geological, or synthetic). The model material exhibits viscous-like response under uniaxial compression, while its behavior under Brazilian testing (diametral compression) is brittle. Despite this feature, crack growth in the samples under tensile stress is attested as semiviscous, although their fracture surface morphology is close to the brittle transcrystalline fracture. It should be noted that human dentin exhibits similar behavior at the same loading conditions [17]. It should be especially noted that cleavage crack growth in highly plastic FCC-metal iridium leads to similar fracture surface morphology [18].

It seems to be that described above difference between deformation behavior of tooth enamel under uniaxial compression at 77 K and room temperature could be caused by the contribution of bioorganic component of enamel. At room temperature the rectilinear course of the compression curve on the first stage is the sum of linear elastic contribution of both inorganic and bioorganic components of tooth enamel, whereas the changing in the inclination angle of the curve on the second stage is the manifestation of viscous (non-linear) response of bioorganic component. Indeed, non-linear elastic behavior under compression occurs in some viscoelastic polymers after 5-7% of deformation [19]. However, the bending of compression curve disappears under testing of enamel in liquid nitrogen, when the viscosity of a bioorganic phase should be absent, and it begins behaving like a rock material. It should be noted that usually compression stress under chewing of normal food does not exceed 50-60 MPa and, hence, this feature of deformation behavior at room temperature of tooth enamel under compression is not critical for teeth. However, the increase of enamel strength with lowering temperature can lead to the critical difference in mechanical properties between enamel and dentin and, as a result, to failure of dentinoenamel junction in a tooth, especially under tensile stress.

The bending of deformation curves under Brazilian testing for some rocks (granite and jasper) and plasma-sprayed Al₂O₃, which, however, is absent in the samples

of human tooth enamel, could be caused by the collapse of pores or other discontinuities and appearance of cracks that begins on the final stage of loading. Indeed, it is reasonable cause for non-linear elastic behavior in solids possessing heterogeneity of structure [20].

The feature of cracking of the sample under Brazilian testing at room temperature is another contribution of bioorganic component into deformation behavior of tooth enamel. It is clearly visible in Fig. 7 that there are many satellite cracks ahead of main crack in the sample under diametral compression. At that, some of them lay with the inclination to the compression axis but have a trend to move parallel to the main crack direction. It is known that crack in tooth enamel usually grows between such structural elements of enamel as the enamel prisms, where bioorganic phase is situated, while it could change the growth direction under external stress influence [1,5]. Indeed, short satellite cracks are initially oriented along the enamel prisms, whereas their further growth, including junction with the main crack, happen along the compression axis. In the model rocks and plasma-sprayed Al₂O₃, the growth of satellite cracks parallel to the compression axis occurs at the contact points of the sample and the hard plate of a testing facility only where stress-distribution is more complicated than in the middle part of the sample.

5. CONCLUSIONS

Deformation behavior of the human tooth enamel was compared with behavior of rocks (granite, serpentinite, and jasper) and plasma-sprayed Al₂O₃ under compression and tension at room temperature. It was shown that tooth enamel and other model materials exhibit the semiviscous-like deformation behavior under compression, whereas their macroscopic response become brittle under tensile stress. Fracture surface morphology was attested as brittle in all model materials, although cracks in them advance by semiviscous manner as a crack in ductile metals. The contribution of viscous bioorganic component in deformation behavior of tooth enamel is detected at room temperature only because bioorganic phases lose their viscosity at low temperatures. However, this contribution does not change the character of deformation behavior of human tooth enamel on the macroscopic scale. The findings obtained here have shown that macroscopic deformation behavior of inorganic solid and minerals does not depend on their genesis (biological, geological or synthetic). In addition, it was confirmed that their behavior strongly depends on the applied loading geometry.

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Общие черты деформационного поведения эмали зубов человека и горных пород

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Аннотация. Обсуждается вклад биоорганических компонентов в деформационное поведение эмали зубов человека. На образцах, вырезанных из эмали, проводили испытания на одноосное сжатие и бразильское испытание (диаметральное сжатие) в жидком азоте (77 K) и на воздухе при комнатной температуре. Проводили сравнение с деформационным поведением некоторых горных пород (гранита, серпентинита и яшмы) и плазменно напыленной керамики Al₂O₃ при сжатии и бразильских испытаниях на воздухе при комнатной температуре. Показано, что эмаль и модельные горные породы демонстрируют квазивязкое деформационное поведение при сжатии, тогда как их макроскопическая реакция становится хрупкой при растяжении. Морфология поверхностей изломов была признана хрупкой во всех модельных материалах, хотя во всех них трещины развиваются на вязкий манер как трещина в пластичном металле. Вклад вязкого биоорганического компонента в деформационное поведение эмали выявляется только при комнатной температуре, поскольку биоорганический компонент теряет свою вязкость при низких температурах. Однако этот вклад не приводит к изменению характера деформационного поведения эмали зубов человека по сравнению с модельными горными породами.

Ключевые слова: эмаль зубов человека; горные породы; одноосное сжатие; диаметральное сжатие; деформационное поведение