

# Hen Eggshell Based Composite as Prospective Dental Material

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## Abstract

Deformation behavior under uniaxial compression and diametral compression of samples compacted from hen eggshell powder with different binders was examined. Depending on concentration and mechanical properties of a binder, deformation behavior of compacted powder can be changed from that inherent to biominerals, for example, tooth enamel and some magmatic rocks, to behavior that close to the viscous behavior of human dentin and some filled polymers or rubber. Hence, mechanical properties of this composite are qualitatively closed to the hard tissues of human tooth. Analysis of dangerous cracks morphology in composites from hen eggshell has confirmed that some features of the viscous deformation behavior are really take place in these materials under compression and tension. This allows considering hen eggshell based composites as prospective materials intended for tooth implants.

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*Keywords:* Hen eggshell; Compacted powder; Binder; Deformation behavior; Cracks

## 1. INTRODUCTION

It is well-known that a restorative material for dentistry should exhibit both high biocompatibility with living organisms and deformation behavior closed to human tooth hard tissues (dentin and enamel) [1–3]. At present time, sintered powder of ZrO<sub>2</sub> ceramics is used as structural material for dental veneeres, whose biochemical and mechanical properties, however, needs to be improved, despite its workability and price meeting the requirements for modern dental restorative materials. Indeed, the phase structure of ZrO<sub>2</sub> ceramics could degrade during a lifetime of implants, while its mechanical properties are higher a little bit than mechanical properties of the tooth enamel. The latter feature induces damage on native teeth around implant while chewing food. Therefore, search for prospective restorative materials for implants and veneers is a relevant problem for dental materials science. The main conditions for such prospective dental material are: (1) the high biocompatibility with human body; (2) its mechanical properties should be a little bit lower than those of tooth hard tissues (dentin and enamel); (3) its price should allow using it in public healthcare.

Veneer on a tooth implant substitutes a crown part of human tooth and therefore its mechanical properties

should be close to a tooth enamel, which is a mineral of biological genesis consisting of 95% calcium hydroxyapatite crystallites and 5% of bioorganic and water. According to the chemical content, the tooth enamel is a covalent solid and, hence, it should be practically nondeformable hard tissue [4,5]. On the other hand, the hard basis of human teeth is dentin consisting of bioorganic matrix strengthened by both nanosized crystallites of calcium hydroxyapatite and a network of collagen fibers; therefore, its deformation behavior should be close to a viscous solid like, for example, a filled polymer or a rubber [6–8]. This clear contradiction is canceled or could be ignored because mechanical properties of tooth hard tissues (tooth enamel and dentin) are the same under loading conditions that appears under chewing of normal human meal [5,9,10]. Obviously, well-known structural materials, such as metallic alloys, ceramics and filled polymers, cannot exhibit mechanical properties of ceramics and strengthened rubber, simultaneously. However, modern composite materials are promising to solve this important problem.

The hen eggshell is a biomineral based on calcium carbonate possessing complicated multiscale morphology meets the main condition for dental restorative materials, namely, the high biocompatibility with living organisms [11]. Recent study has shown that mechanical prop-

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erties of samples cut from hen eggshell also meet other conditions for dental restorative materials, because they are lower than properties of tooth enamel, but could be suitable for chewing food [12]. Another important property of hen eggshell is its ability to effectively suppress crack growth [13]. Indeed, dangerous macroscopic crack in eggshell sample can be grown up under bending. Taking into account that hen eggshell is a covalent solid, this ability looks fantastic. In addition, hen farms can supply needed quantity of certified eggshell at appropriate price.

In its native state, hen eggshell cannot be used for manufacture of dental veneers for tooth implants because of its shape (thin plate with a complicated geometry). However, the powder metallurgy technology that includes the grinding of an initial raw material into powder and its further compaction with different binders, could allow the usage of hen eggshell as the structural material for veneers in dental implants. The aim of this work is to check this supposition, namely, to manufacture the samples from compacted hen eggshell with different binders and to examine their deformation behavior under compressive and tensile load including fracture behavior.

## 2. MATERIALS AND METHODS

Biological material for research (white and brown colored hen eggs of C1 category, according to the Russian Standard) were purchased in supermarkets of Yekaterinburg. Hen eggs were washed in warm water and were dried in air. Two holes of 2 mm in diameter were drilled in each egg for the extraction of liquid bioorganic matter. After that inner surfaces of eggs were washed by warm water and dried in air during a few days. Eggshell was milled in an electric coffee grinder. The size of hen eggshell particles prepared with the help of this milling device was few hundred micrometers. Technological experiments have shown that such size of powder particles guarantees that samples are homogeneous with minimal level of surface defects. Therefore, no advanced types of laboratory milling devices that allow obtaining the smaller sized powders were used in this work.

Samples for mechanical testing with either circular cross-section 12 mm in diameter or square cross-section  $10 \times 10$  mm were compacted from both white and brown eggshell powders using steel press-forms under pressure of 200 bar. The following glues were applied as binding substance: (1) polymer rubber glue, (2) polymer PVA glue, (3) silicon glue for paper, which were purchased in supermarkets of Yekaterinburg. The choice of binders was caused by following reasons. First, these glues have well-known and stable mechanical properties: rubber glue exhibits the high plastic and adhesive properties; PVA glue is also plastic substance, but solute in water; while silicon glue for paper is brittle substance. Second, these binders are widely

available substances. Third, they have low price. Naturally, none of them can be applied in clinical experiments, but this kit of glues a priori covers wide range of mechanical properties of binders. Three sets of cylindrical samples with different diameter to height ratios (2:1, 1:1 and 1:2) were prepared. Two sets of cuboid samples with sizes of  $10 \times 10 \times 5$  mm and  $10 \times 10 \times 10$  mm were also prepared for comparison. The main part of mechanical tests was carried out on the tablet-shaped samples with the ratio of 2:1 and cuboid ones with the size of  $10 \times 10 \times 5$  mm, whose mass was 1.4 g plus 0.1 g of binding glue (Fig. 1). In addition, one set of samples was compacted without bonding substance, whereas another set was compacted with 0.3 g of rubber glue as bonding agent. No morphological difference was detected between the samples prepared from white and brown eggshell, but brown particles give better optical contrast than the white ones. Therefore, the major volume of tests was carried out on the samples compacted from brown eggshell.

Mechanical testing was carried out at room temperature in air using two deformation schemes: uniaxial compression and diametral compression sometimes called as Brazilian test. Shimadzu™ Autograph AG-X 50kH (traverse rate of 1 mm/min) testing machine was used, while experimental data were processed with the help of Trapezium™ software. The set from ten samples was used to examine every type of compacted eggshell powder. Prior and after testing each sample was documented with an optical microscope under magnification of  $\times 10$ . Cracks appearing in the samples under loading were studied with a metallographic microscope under magnification of  $\times 50$ .

## 3. RESULTS

Engineering deformation curves under uniaxial compression for both tablet and cuboid samples were plotted. Experiments have shown that the shape effect is observed in both tablet and cuboid samples (Fig. 2). As it was expected, the best and stable mechanical properties were exhibited by “thin” samples, whereas mechanical behavior of “thick” samples was unstable because of cracking of their edges. The typical compression curves for compacted hen eggshell powder samples (tablets of 12 mm in diameter and 6 mm in height and cuboids of  $10 \times 10 \times 5$  mm) with different binders are shown in Figs. 3a and 3b, respectively. Loading of samples was stopped as soon as bend appeared on the engineering curve meaning start of cracking in the sample or the beginning of its failure. Elastic modulus of sample was calculated using an inclination angle of the deformation curve on its linear stage with a help of Trapezium™ software. Value of compression stress at the point of bending of deformation curve is treated as the ultimate compression stress for material, whereas the deformation at this point is accepted as the maximal deformation or the

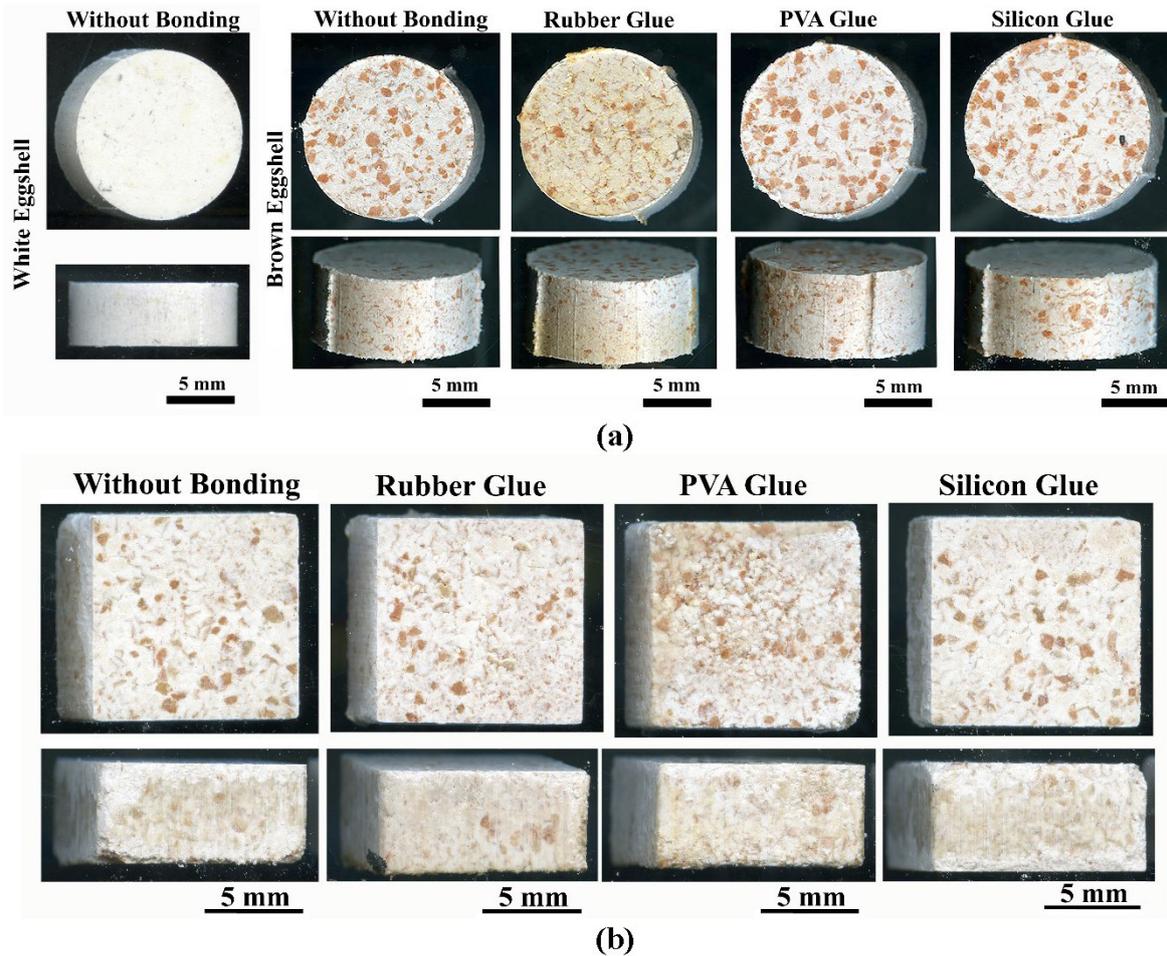


Fig. 1. Samples for mechanical testing prepared from compacted brown hen eggshell with different bonding agents: (a) tablet samples (12 mm in diameter and 6 mm in height), (b) cuboid samples (10×10×5 mm).

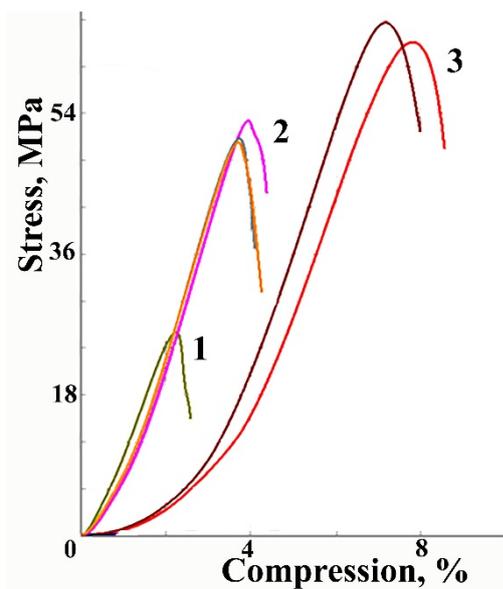
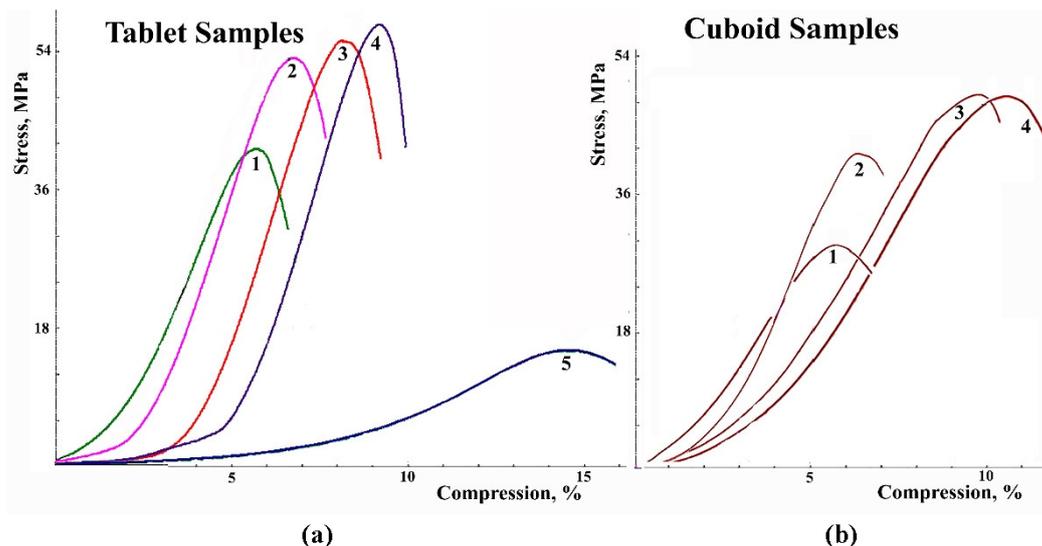


Fig. 2. Deformation curves for uniaxial compression of tablet samples of compacted hen eggshell powder without binder: curve 1 – diameter to height ratio is 2:1; curves 2 – diameter to height ratio is 1:1; curves 3 – diameter to height ratio is 1:2.

deformation prior to failure of material [14]. These mechanical characteristics of tested materials are presented in Table 1. No difference in deformation behavior between samples compacted from white and brown hen eggshell powders were detected.

Engineering compression curves of the samples containing small quantity of binder (approximately 0.1 g of different glues) can be approximated by a straight line at least in their middle part prior to their bending, where elastic moduli are determined. The maximal compression stress depends on the properties of binder. It is about 45 MPa for plastic rubber glue, while for other binders it grows to 50–60 MPa. Such type of deformation behavior would be considered as brittle, which is inherent to covalent materials [15]. However, the value of deformation prior to failure (which is at least 5%, but can reach up to 10%) is too high for brittle deformation behavior [16]. In addition, the samples never separate in small fragments after the bending of deformation curve, as it occurs in silica glasses and silicon crystals [17]. Qualitatively type of deformation behavior of the model materials under uniaxial compression did not depend on the shape of samples,



**Fig. 3.** Deformation curves for uniaxial compression of samples of compacted hen eggshell powder with different binders having (a) cylindrical shape (12 mm in diameter 6 mm in height) and (b) cuboid shape (10×10×5 mm). Samples were prepared from compacted brown hen eggshell with different bonding agents: curve 1 – polymer rubber glue (0.1 g), curve 2 – polymer PVA glue (0.1 g), curve 3 – without binder, curve 4 – silicon glue (0.1 g); curve 5 – polymer rubber glue (0.3 g).

**Table 1.** Mechanical properties under uniaxial compression and diametral compression (Brazilian test) of compacted hen eggshell powder with different binders.

Uniaxial compression	Tablet samples	No.	Binder	Elastic modulus, GPa	Ultimate compression stress, MPa	Deformation prior to failure, %
		1	Polymer rubber glue (0.1 g)	0.9	45	5
2	PVA glue	1.0	50	7		
3	Compacted without binder	1.0	52	9		
4	Silicon glue	1.0	60	10		
5	Polymer rubber glue (0.3 g)	not determined	15	15		
Uniaxial compression	Cuboid samples	No.	Binder	Elastic modulus, GPa	Ultimate compression stress, MPa	Deformation prior to failure, %
		1	Polymer rubber glue	0.8	30	6
		2	PVA glue	1.1	40	6
		3	Compacted without binder	1.0	48	10
		4	Silicon glue	1.0	48	12
Diametral compression	No.	Binder	Elastic modulus, GPa	Ultimate stress, MPa	Deformation prior to failure, %	
	1	PVA glue	1.3	7.5	0.5	
	2	Silicon glue	1.0	7.0	1.0	
	3	Polymer rubber glue (0.1 g)	1.1	3.5	1.0	
	4	Polymer rubber glue (0.3 g)	not determined	0.2	3.5	

although the maximal stress for cuboids was lower than that for tablets. Working or contact surfaces of both tablet and cuboid samples after compression testing are shown in Figs. 4a and 4b, respectively. It is visible that cracks appear on the edges of samples. According to mechanics of deformed solids, cracks in the samples propagate in the areas with maximal cleavage stresses. Therefore, their trajectories repeat the round edge in the tablet samples, while cracks cleave triangular fragments in the cuboid samples.

The increase of the plastic rubber glue concentration from 0.1 g to 0.3 g leads to qualitative change in the deformation behavior of compacted hen eggshell powder (Fig. 3a, curve 5). It becomes close to such filled polymer as rubber [18]. Indeed, the ultimate compression stress drops by 3÷4 times, deformation prior to failure reaches 15%, while the value of elastic modulus cannot be detected by Trapezium™ software.

Engineering deformation curves of the model materials subjected to Brazilian testing (tablets of 12 mm in

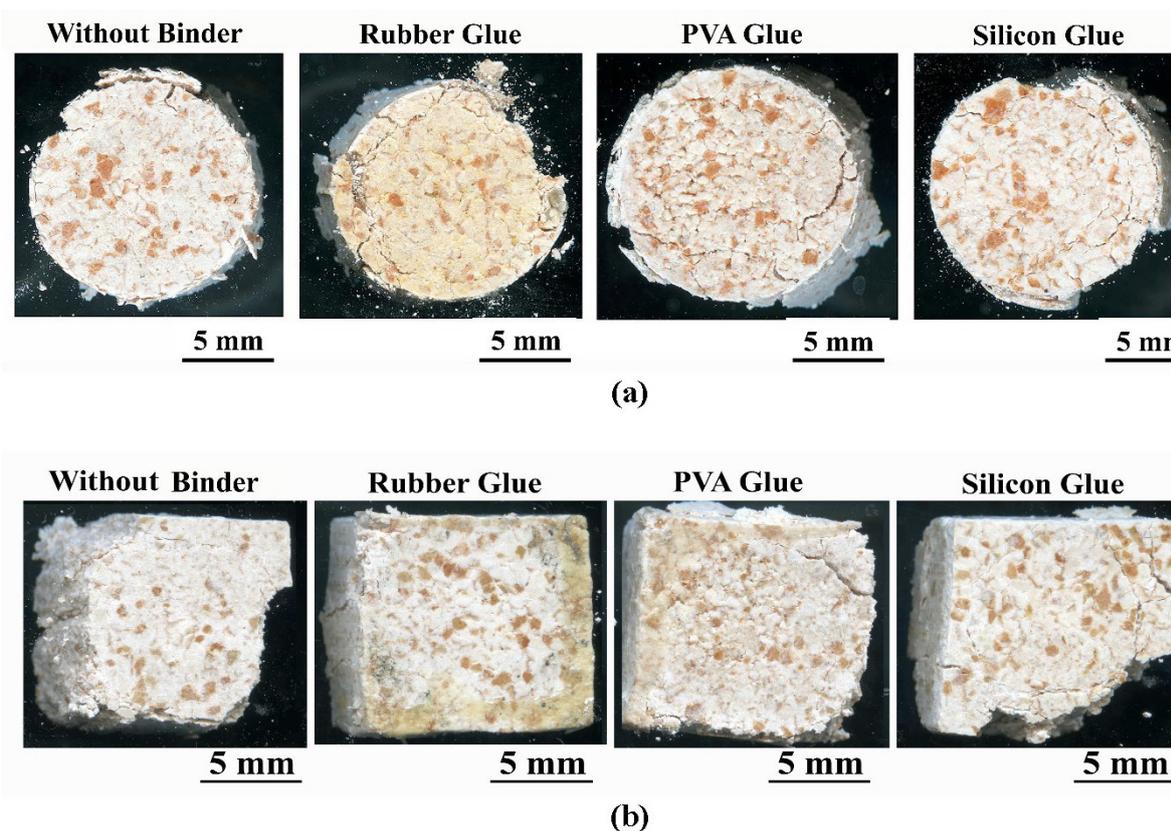


Fig. 4. Working/contact surfaces of samples prepared from compacted brown hen eggshell with different binders (without binder; rubber glue; PVA glue; silicon glue): (a) tablet shape; (b) cuboid shape.

diameter and 6 mm in height) are presented in Fig. 5, while their mechanical properties are given in Table 1. The deformation curves for materials that contained about 0.1 g of binder can be approximated by a straight line, and the deformation prior to failure for all materials was less than 1%. Hence, the model materials exhibit the brittle deformation behavior under applied tensile stress [14,15,19].

In spite of this, the samples never cleave under loading like brittle materials such as silica glasses or silicon crystals. The cracks in the samples that appeared under Brazilian testing are shown in Fig. 6. They look like thin broken lines crossing the samples along the axis of compression. Their trajectory is determined by maximal cleavage stress, which is situated between the points of contact of the hard plates of testing machine and tablet sample.

Similar to the case of uniaxial compression, the increase of plastic binder (polymer rubber glue) concentration from 0.1 g to 0.3 g causes the change in deformation behavior, which becomes close to behavior of filled polymer or rubber (Fig. 5, curve 4). The ultimate stress drops by one-two orders of magnitude, while deformation prior to failure grows up by few times, while the engineering curve runs approximately parallel to the deformation axis. Trapezium™ software could not determine the value of elastic modulus of these materials as well.

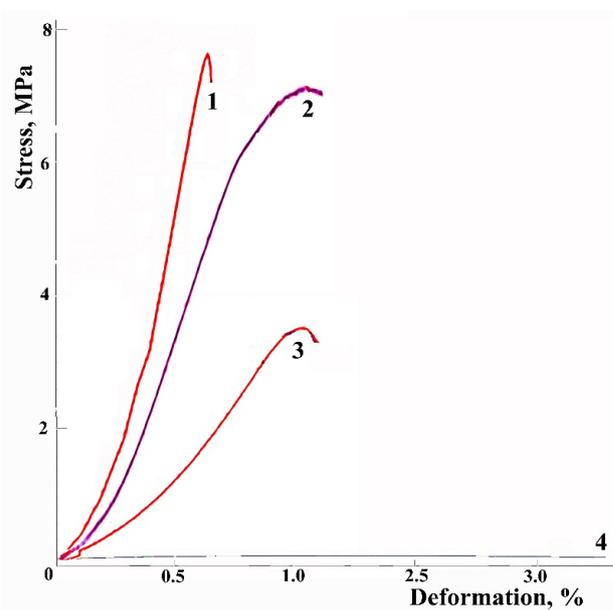


Fig. 5. Deformation curves for diametral compression (Brazilian test) of samples of compacted hen eggshell powder with different binders: curve 1 – polymer PVA glue (0.1 g); curve 2 – silicon glue (0.1 g); curve 3 – polymer rubber glue (0.1 g); curve 4 – polymer rubber glue (0.3 g).

Metallographic examination of the dangerous cracks in the samples under Brazilian testing at high magnifica-

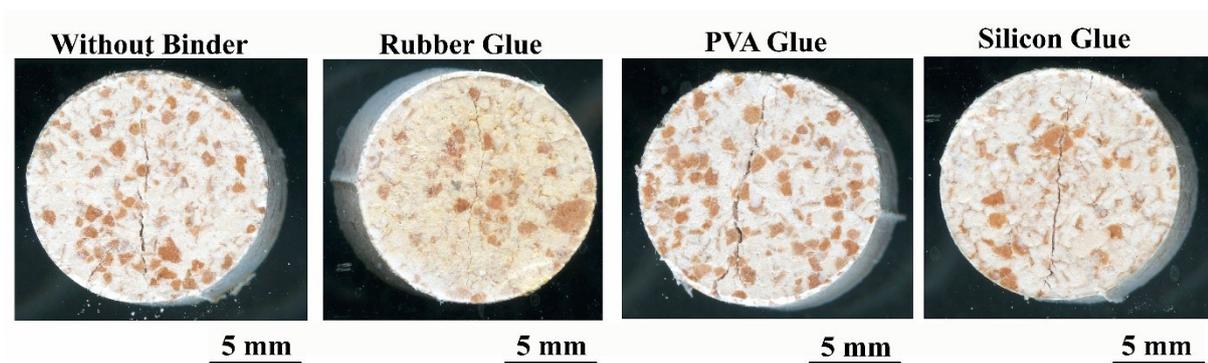


Fig. 6. Working surfaces of samples prepared from compacted brown hen eggshell powders with different binders (without binder, rubber glue, PVA glue, silicon glue) after Brazilian testing.

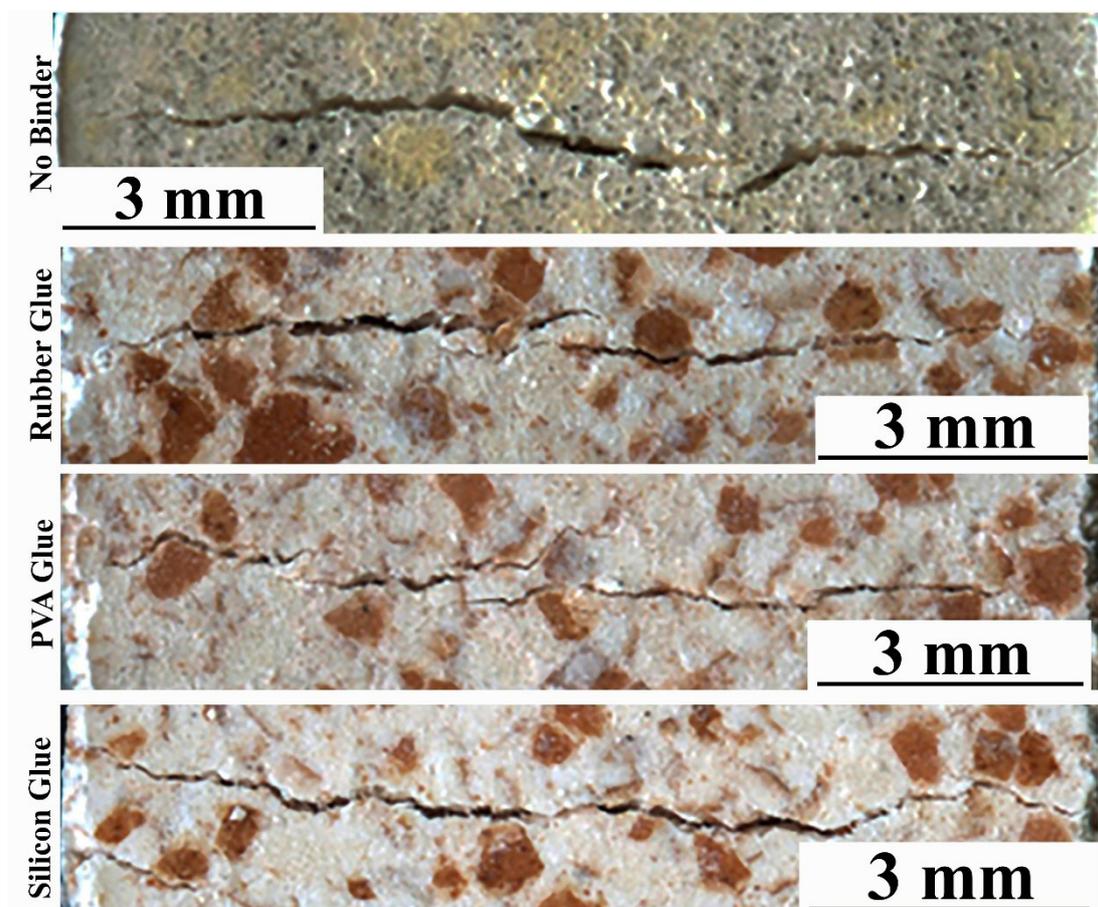
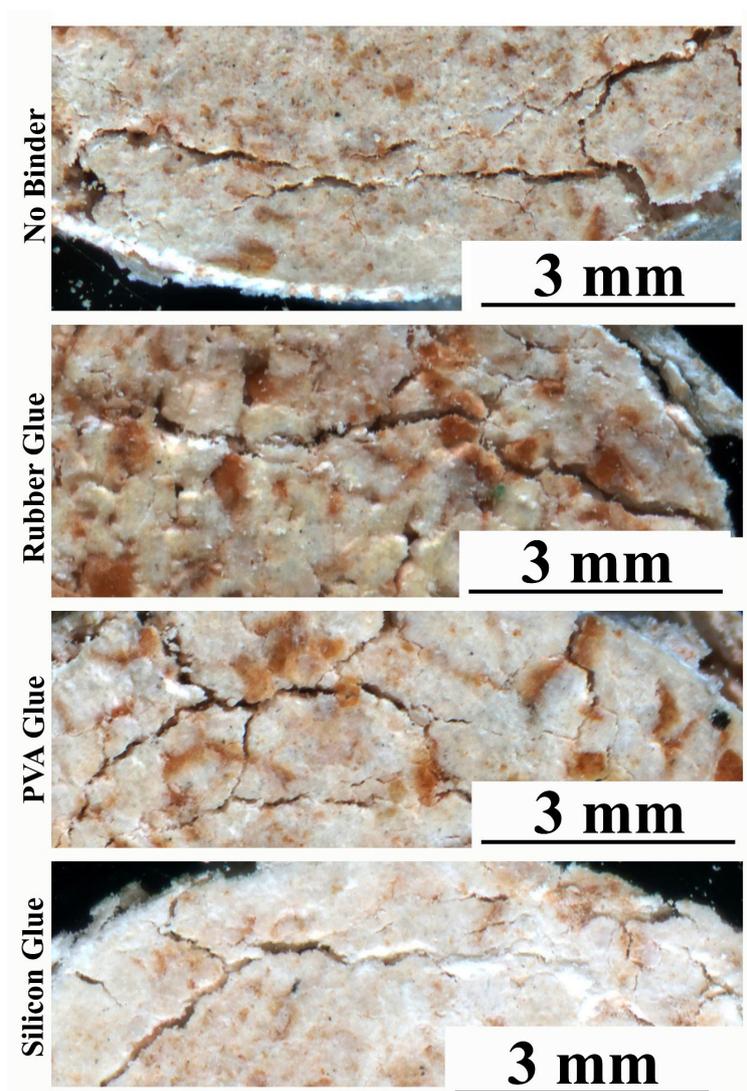


Fig. 7. Dangerous cracks on the working surfaces of samples prepared from compacted hen eggshell powder with different binders: without binder (white eggshell), rubber glue (brown eggshell), PVA glue (brown eggshell), silicon glue (brown eggshell).

tions has shown that they consist of small cracks, which have a trend to merge with each other (Fig. 7). As a rule, these cracks are found in the vicinity of the straight line connecting the points of contact between the sample and traverses of testing machine. No differences caused by different binders were detected. Comparison of cracks in the model materials with cracks in typical brittle solids like, for example, silicon crystals, allows concluding that there are many differences between them [16,17]. Major-

ity of such cracks in the model materials possesses sharp tips, however, many of them have considerable angle of opening and pore-like shape. It seems their morphology is rather close to cracks in the neck region of a ductile metal than cracks in a brittle solid [20,21]. This paradoxical conclusion is supported by the fact that the model materials never cleave under Brazilian testing when the loading is removed after the bending point on the engineering curve.



**Fig. 8.** Cracks on the contact surfaces of samples prepared from compacted hen eggshell powder with different binders after uniaxial compression: without binder; rubber glue; PVA glue; silicon glue.

Analysis of crack morphology in the tablet samples under uniaxial compression did not discover any differences on the microscopic scale with previous case. Long cracks consist of small pore-like cracks, but their macroscopic trajectories are more complicated than under Brazilian testing (Fig. 8). Indeed, the stress distribution near the sample edge under uniaxial compression is more complicated than under diametral compression.

#### 4. DISCUSSION

Analysis of deformation behavior of the model materials under uniaxial compression and Brazilian testing has shown that samples compacted from hen eggshell powder with or without binder exhibit two different types of behavior. Compacted eggshell powder samples with no binder or with small amount of glue (0.1 g per 1.4 g) behave like rocks or biominerals, including human tooth

enamel [13,22]. Increasing amount of plastic rubber glue up to 0.3 g per sample induces the change in deformation behavior so it becomes close to behavior of filled polymers, including human dentin. Hence, varying concentration of binder we can control deformation behavior of biocomposite in the wide range from rock to rubber. This is very important for its application in dentistry, because human tooth is a natural composite structure consisting of elastic but strong dentin covered by tooth enamel or calcium apatite based biomineral. Maximal compression and tensile stress of the model materials is considerably lower than that of human tooth hard tissue. However, it is quite enough for chewing human food by dental structure made from compacted eggshell powder without a risk of damaging neighboring natural teeth and jaw bones. Indeed, it is much easier to simply replace composite veneer on tooth implant than to heal damage of natural teeth and jaw fractures. Another advantage of hen eggshell is its high bio-

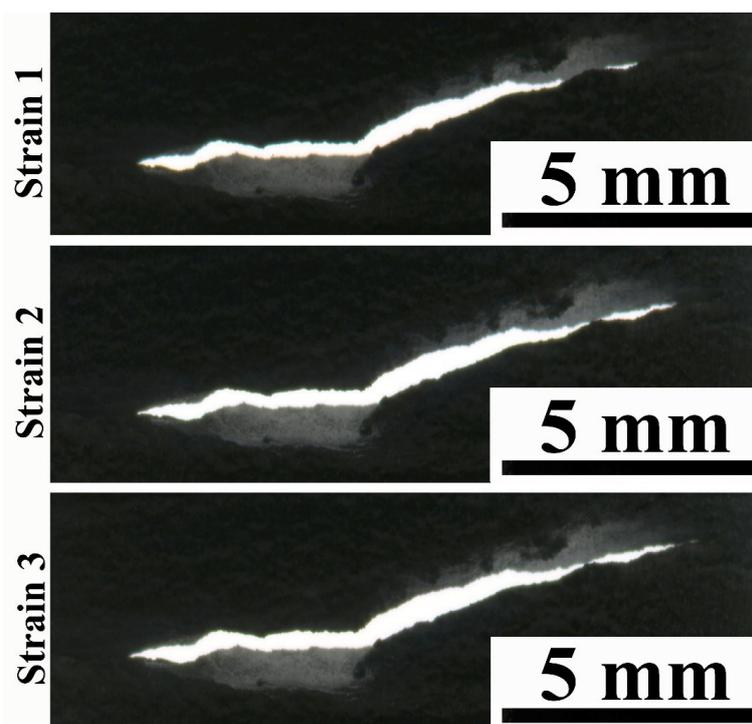


Fig. 9. Growth of dangerous crack in neck region of plane aluminum polycrystalline sample under tension.

compatibility with living organisms. Besides, these composites may be appropriate substrate for growth of soft tissues that opens new prospects for dental orthopedics, namely, for joining of gum soft tissues with dental implant in human mouth. Modern additive technologies open certain prospects for elaboration of synthesis techniques of the hen eggshell based composite structures having dentin-like basic properties, but covered with enamel-like material.

It is well known that study of cracks provides an opportunity to obtain an information on mechanisms of stress accommodation in material on the microscopic scale. This approach may be useful in the case of hen eggshell composites. Indeed, morphology of dangerous cracks in the model materials looks different from cracks in brittle solids [15–17]. However, it is similar to dangerous cracks in elastic dentin and tooth enamel, where the main crack propagates due to merging of pore-like cracks in the plastic zone in front of crack tip [3]. In addition, this crack growth mechanism looks similar to dangerous crack growth in neck region of a plane aluminum sample under tension (see Fig. 9). In other words, deformation behavior of the hen eggshell composite on the microscopic scale displays some elements of ductile deformation behavior [21]. And this feature of the model materials is not unique because it is also observed in some magmatic rocks and in the samples of bird eggshell under bending [22,23]. Therefore, increase of plastic binder concentration in hen eggshell composite induces the transition from brittle to ductile behavior on the macroscopic scale in them.

## 5. CONCLUSIONS

Deformation behavior of a hen eggshell based composite under uniaxial compression and Brazilian testing is examined. It was shown that mechanical properties and concentration of binder govern the type of deformation behavior of composite, which varies from inherent to rocks and biominerals [19] to filled polymers [18]. Therefore, this feature of hen eggshell based composite allows considering it as prospective restorative material for dentistry, namely, for tooth implants.

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## Композит на основе куриной скорлупы как перспективный ресторативный материал для стоматологии

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**Аннотация.** Изучено деформационное поведение при одноосном сжатии и диаметральном сжатии образцов, спрессованных из порошка скорлупы куриных яиц с различными связующими. В зависимости от концентрации и механических свойств связующего деформационное поведение спрессованного порошка может изменяться от собственного биоминерала, например, зубной эмали, и некоторым магматическим породам, до поведения, близкого к вязкому поведению человеческого дентина и некоторых наполненных полимеров, и резины. Таким образом, механические свойства данного композита оказываются качественно близкими к твердым тканям человеческого зуба. Анализ морфологии опасных трещин в композитах из скорлупы куриных яиц подтвердил, что некоторые особенности вязкого деформационного поведения при сжатии и растяжении здесь действительно имеют место. Это позволяет рассматривать композиты на основе скорлупы куриных яиц в качестве перспективных материалов для стоматологии, предназначенных для зубных имплантатов.

**Ключевые слова:** скорлупа куриных яиц; компактированный порошок; связующие материалы; деформационное поведение; растрескивание