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On Some Feature of Deformation Behavior of a Bird Eggshell

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Abstract

Behavior of bird eggshells—biominerals consisting of about 90% of calcium carbonate—under bending is examined. Eggs of several birds were used with two sets of samples cut from every bird eggshell. Mechanical testing was carried out on dry and wet samples in air. Cracks in eggshell samples under bending were studied in situ using a light microscope. Eggshell exhibits brittle behavior on the macroscopic scale in both dry and wet states. However, the crack width could be increased by increasing bending deflection similar to ductile metals under tension. Wet samples show lower bending strength. The morphology of cracks in an eggshell under bending is close to the crack in neck region of a flat aluminum sample. It may be concluded that a bird eggshell under bending exhibits some features of ductile fracture on the microscopic scale.

Keywords: Biomineral; Bird eggshell; Bending; Deformation; Fracture

1. INTRODUCTION

Since ancient times, nature was the source of ideas that promote an advance in structural materials. For example, a copy of morphology of some biological tissues is considered as actual trend in modern materials science, which could provide materials (sometimes called biomimetic) with a unique kit of mechanical properties [1,2]. However, the question how hierarchically organized structure of a biological tissue makes certain combination of different properties possible in a synthesized material is still open.

Bird egg provides the evolution of a bird from an embryo to a chick due to protection by its eggshell. Basic structural element of an eggshell is calcium carbonate of the biological genesis that could be called a mineral compound similar to a rock material. It is inorganic matter that possesses the covalent chemical bonding and, hence, it must exhibit the brittle deformation behavior. The mechanical properties of bird eggshells were extensively examined by many researchers [3]. The majority of these works have studied the behavior of an egg in whole as an object for application of mechanical loading [4]. It was shown that egg exhibits high strength although eggshell being prone to the brittle fracture [5,6]. Besides, eggshell is a unique hard tissue because it possesses two natural surfaces, which remain the same during preparation of samples for mechanical testing. This circumstance may be important for understanding the features of its deformation behavior in comparison with other rock materials and minerals including tooth enamel.

It sounds quite unusual, but mechanical properties of samples cut from an eggshell were not studied in detail before. Our experiments have shown that samples cut from hen eggshell behave in the brittle manner under three-point bending both in air and water [7,8]. However, dangerous cracks in them could start growing when the bending deflection increases that is not inherent to the brittle behavior of a solid. Is it a feature of its biological genesis, when the structure of calcium carbonate includes bioorganic inclusions? Or is there another cause? The aim of this work is the detail examination of deformation behavior of bird eggshells under bending both in air and water including crack growth under in situ experiments in a metallographic microscope.

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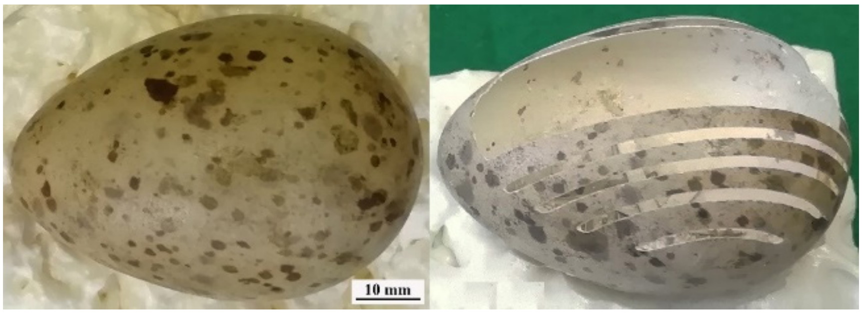
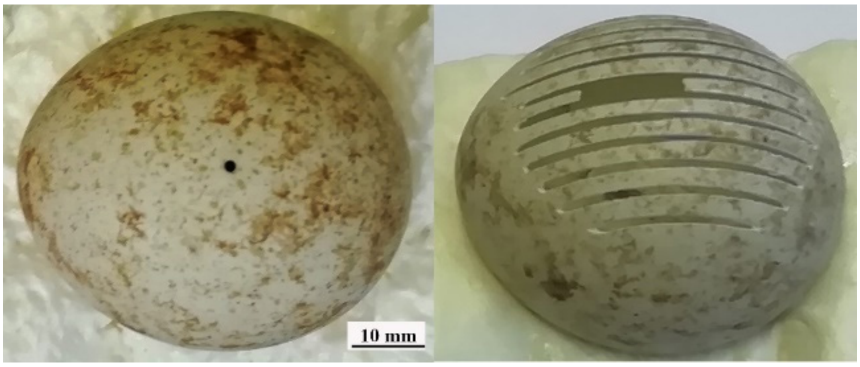
2. MATERIALS AND METHODS

Eggs of several wild and domestic birds that were used as the experimental biological material are: (1) black-headed gull (larus ridibundus), (2) buzzard (buteo buteo), (3) capercaillie (tetrao urogallus), (4) gray heron (ardea cinerea), (5) goose (anser domesticus) and (6) hen (gallus domesticus). They are shown in Figure 1. Hen eggs (standard and giant) and goose eggs were obtained from a retail outlet in Yekaterinburg. The soft biological tissues of hen and goose eggs were extracted from eggshells before preparation of samples for mechanical testing. Eggshells of the wild bird eggs were kindly supplied by Dr. Svetlana Meshcheryagina from the Institute of Plant and Animal Ecology of the Russian Academy of Sciences at Yekaterinburg.



(a)



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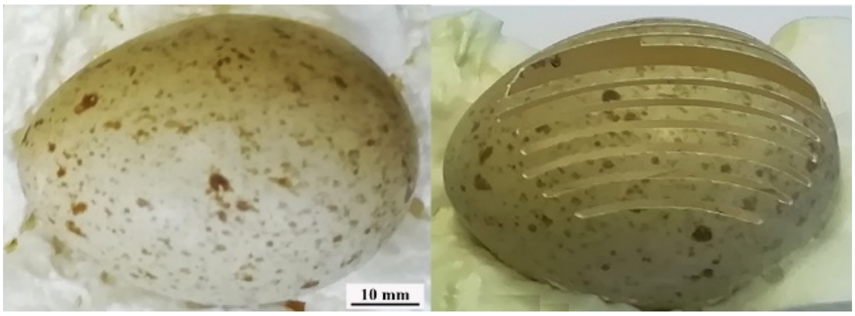

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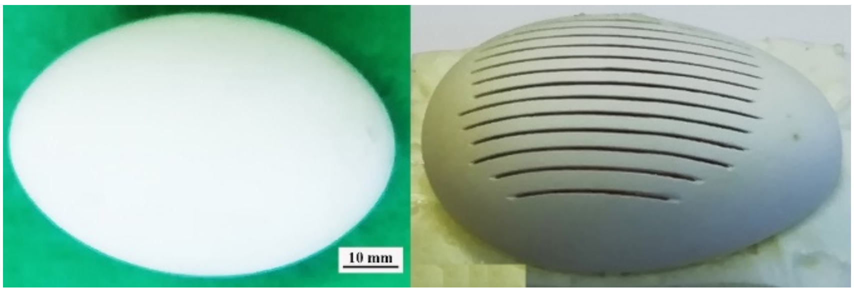

(d)

Fig. 1. Samples for bending cut from bird eggshells: (a) black-headed gull; (b) buzzard; (c) capercaillie; (d) goose.

An empty egg was mounted on the substrate made from polyurethane foam purchased in a construction supermarket. This procedure guarantees powerful fixing and integrity of an eggshell on the cutting device equipped with a diamond disc saw (thickness 0.1 mm). Some important details of sample preparation are given in Refs. [7,8].

Samples with a width of 2–3 mm were cut from the middle part of an eggshell along an egg axis (Fig. 1). The thickness of samples varied from 0.3 mm to 0.5 mm depending on a bird kind. The samples in the undeformed state are shown in Fig. 2. Three-point loading was chosen as the deformation scheme for mechanical testing. Shimadzu™ AG-S 2kN testing machine was used in experiments. Rate of movement of the traverse was 0.1 mm/min. Shimadzu's 3-point bending device was used for testing. The distance between the lower prisms of bending device was 10 mm. The length of the samples was about 15–20 mm. Shimadzu software Trapezium™ was used for processing the data. Two sets of samples from every bird eggshell (10 pieces in each one) were prepared for the mechanical testing. The first one contained dry samples, while the second one contained samples exposed to water for 24 hours. Morphology of fracture surfaces of tested samples was examined with scanning electron microscope Tescan Vega LMS.

Crack evolution in the samples under bending was studied during in situ experiments on metallographic microscope equipped with a digital camera. The loading geometry accepted for the experiment was close to the geometry of three-point bending used in the Shimadzu device. The distance between the lower prisms was 10 mm, as well. The bending deflection was set with a micrometer in increments of 10 μm. The working surfaces of samples were preliminary painted in violet color to increase the contrast of images.

The following testing procedure was used. The sample was mounted by curved side over an objective of metallographic microscope. The bending of sample was applied by means of increasing the bending deflection until the crack appeared. The area in vicinity of crack was documented under magnifications of x100 and x500. Then the procedure was repeated. As a rule, majority of samples withstood two bendings when the crack appeared and grew up. Topograms of the areas in the vicinity of the cracks were obtained from the digital images of the working surfaces. The morphology of the cracks were examined using these topograms.

3. RESULTS

The typical deformation curves for the bird eggshells of black-headed gull, capercaillie, gray heron, hen and buzzard in the dry state under bending in air are presented in Fig. 3, while their mechanical properties are listed in Table 1. The engineering curves under bending of goose eggshell in both dry and wet states are shown in Fig. 4. Mechanical properties of the samples in the wet state are also given in Table 1. The common features of deformation behavior of the samples are: (1) tiny deformation prior to failure that varies from 0.2% to 0.5%; (2) the engineering curve could be approximated by a straight line; (3) long time exposition in water does not qualitatively change the type of deformation behavior, while it somewhat decreases its strength and deformation prior to failure. The strength under bending of a bird eggshell varies in the range of 15–30 MPa and it depends on a kind of bird, while strength decrease in the wet state is always in the range of measurement error. Such deformation behavior is estimated as brittle and it is inherent to inorganic non-metallic solids having covalent chemical bonding, for example, ceramics, glasses, minerals and rocks.

Examination of fracture surfaces of tested samples confirms this conclusion inasmuch for all kinds of the birds their morphology was close to the brittle transgranular fracture with the river patterns being the main feature (Fig. 5).

However, there is circumstance that contradicts this conclusion. Experiments have shown that majority of tested samples do not fall apart when an engineering curve passes the bending point, as it should occur in a brittle solid. In so doing, cracks that crossed the sample from edge to edge, are clearly detected on the surfaces of samples, while tested samples attain fractured profile as is visible in Fig. 2. It is surprising, but these samples do not fail during transfer from the testing device to a storage box or to a microscope table for metallographic examination. It means that crack growth in these solids with covalent chemical bonding could be effectively suppressed. Moreover, water environment does not qualitatively change the fracture behavior of the samples cut from a bird eggshell.

This feature of the fracture behavior of a bird eggshell is examined during in situ bending experiments with metallographic light microscope. Crack that appeared under bending in the sample cut from black-headed gull eggshell is shown in Fig. 6. It crosses over curved surface of the sample from edge to edge following complicated broken trajectory. The bending of sample with the deflection of 100 μm causes the growth of crack width by about two times, but does not lead to the failure of the sample. The crack faces are uneven and blurred similar to a pore surface in ductile metals both at the moment of appearance and after additional bending. Fracture behavior under bending of the samples cut from black-headed gull eggshell that were exposed to water during 24 hours is similar as is demonstrated in Fig. 7 with the exception of the value of bending deflection for crack growth initiation, which is about 50 μm. Cracks in the samples cut from eggshells of other kinds of birds behave the same way as shown in Figs. 8–17.

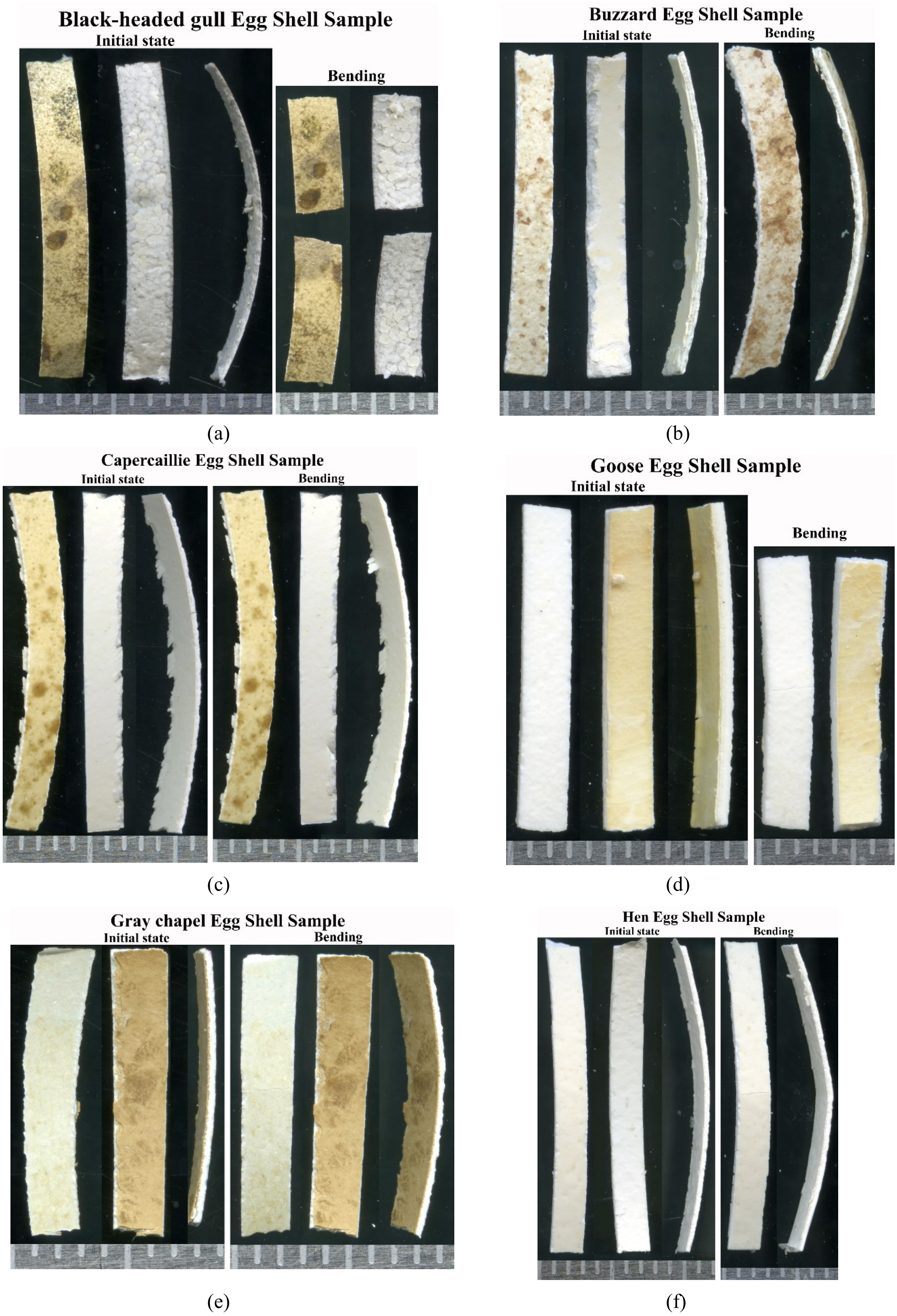

Fig. 2. Samples for bending cut from bird eggshells: (a) black-headed gull; (b) buzzard; (c) capercaillie; (d) goose; (e) gray chapel; (f) hen.

Table 1. Bird eggshells mechanical properties under bending.

Bird eggshell	Effective elastic modulus, GPa	Maximal strength, MPa	Deformation prior to failure, %
Black-headed gull eggshell in air	10±5	17±3	0.2
Black-headed gull eggshell exposed to water	15±5	14±3	0.1
Buzzard eggshell in air	10±3	18±5	0.2
Buzzard eggshell exposed to water	7±3	15±3	0.3
Capercaillie eggshell in air	20±5	34±4	0.2
Capercaillie eggshell exposed to water	16±2	30±5	0.2
Goose eggshell in air	10±3	30±4	0.5
Goose eggshell exposed to water	10±3	24±3	0.4
Gray heron eggshell in air	20±2	20±5	0.1
Gray heron eggshell exposed to water	10±2	20±2	0.2
Hen eggshell in air	15±5	27±7	0.3
Hen eggshell exposed to water	15±5	27±7	0.3

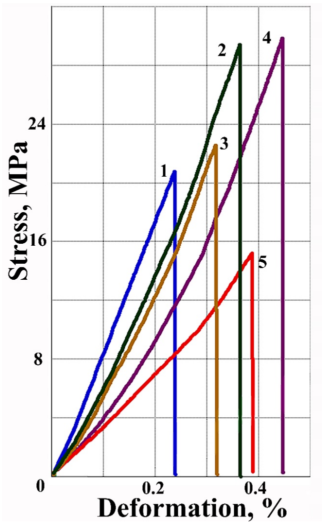

Fig. 3. Deformation curves of the samples cut from bird eggshell under bending in air in the dry state: curve 1 – black-headed gull; curve 2 – capercaillie; curve 3 – gray heron; curve 4 – hen; curve 5 – buzzard.

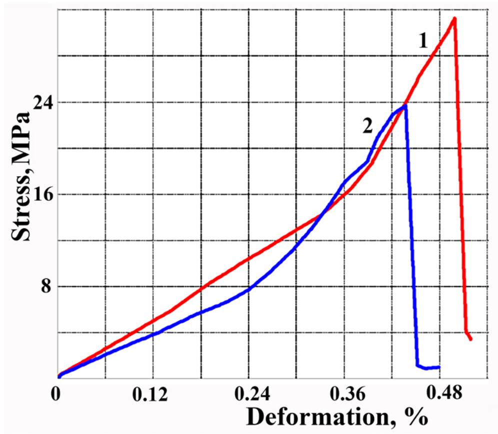

Fig. 4. Deformation curves of the samples cut from goose eggshell under bending in air: curve 1 – dry state; curve 2 – samples exposed to water for 24 hours.

4. DISCUSSION

Obtained findings confirm that a bird eggshell behaves like a brittle solid under bending on the macroscopic scale. It meets with both its covalent type of chemical bonding and its fracture mode, which was attested as the brittle transgranular fracture. However, there are important distinctions between behavior of a bird eggshell and behavior of a brittle solid under bending on the microscopic scale. The first feature is the braking of crack growth under bending of the sample. It displays in the visible increase of crack width under step-by-step bending. In other words, crack growth under bending in a bird eggshell stops when the load is removed and resumes when it is applied again. The second feature is the deformation behavior of a bird eggshell in a water-containing environment. This feature is close to the Rehbinder's effect for a rock, when a liquid environment causes the strength decrease of materials, but without its embrittlement [9]. Indeed, strength of a bird eggshell decreases under bending in water as predicted by the Rehbinder's rule (see Table 1), whereas the morphology of dangerous cracks and morphology of fracture surfaces continue to be the same. It should be noted that values of strength of eggshell under bending in air and in water lay in the range of the measurement error and, therefore, we cannot accurately determine this effect as the Rehbinder's one on the basis of mechanical testing only. However, in situ bending in the metallographic microscope has shown that the bending deflection needed for crack growth initiation in water two times lower than that in air. Hence, the conclusion on the Rehbinder's effect in a bird eggshell is supported by obtained experimental data.

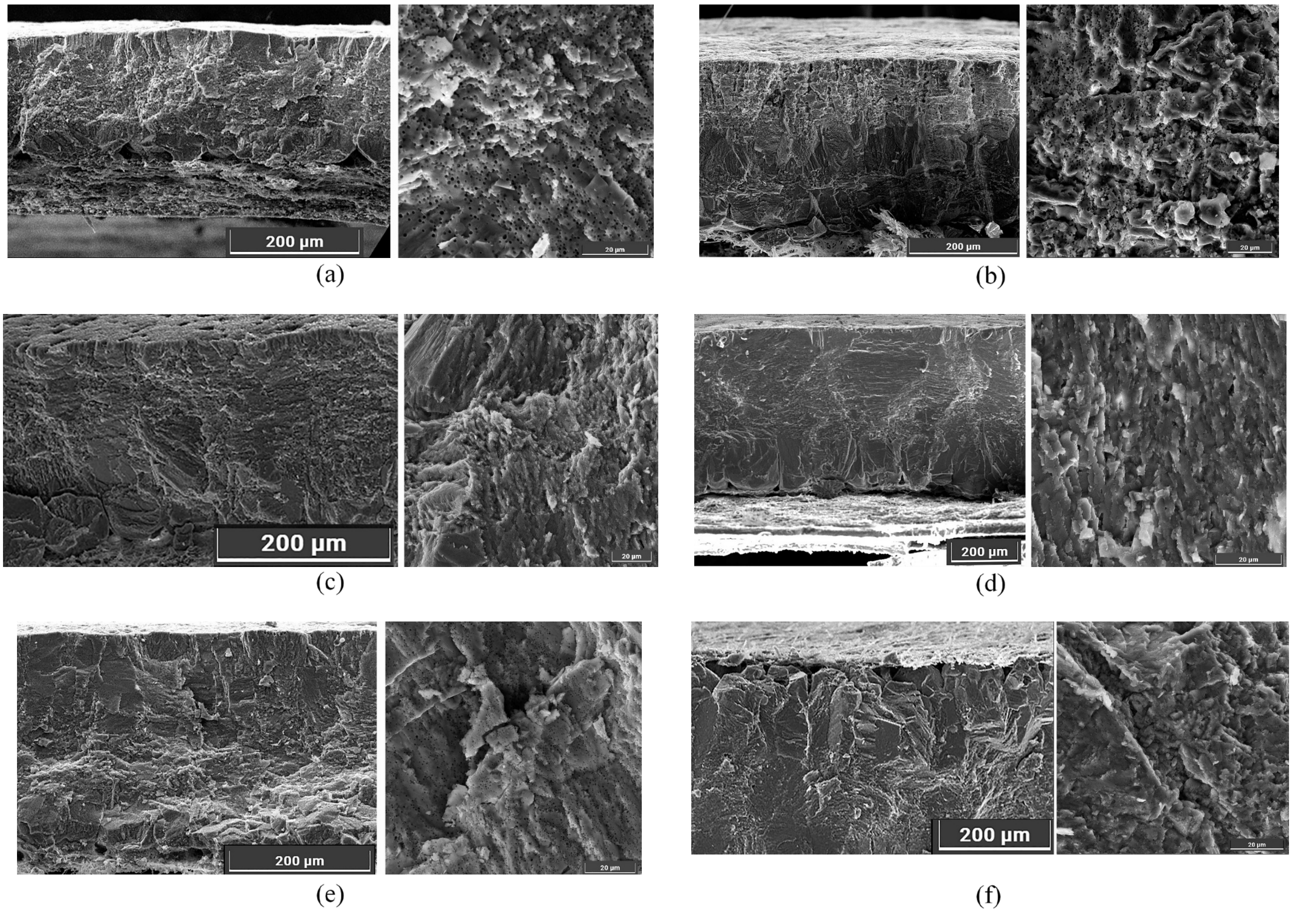

Fig. 5. Fracture surfaces of samples after bending cut from bird eggshells: (a) black-headed gull; (b) buzzard; (c) capercaillie; (d) goose; (e) gray chapel; (f) hen.

Step-by-step crack growth without sample failure during step-by-step loading, is usually observed in a ductile metal under tension. In both types of material, a dangerous crack propagates in an area of localization of irreversible deformation that sometimes is called “neck” or “neck region”, which appears under tensile loading [10,11]. Dangerous crack in the neck region of flat aluminum polycrystalline sample under tension is shown in Fig. 18. It grows in the area with the maximal tensile stress level and possesses a broken profile and rough faces. Comparison of dangerous crack in polycrystalline aluminum with dangerous cracks in minerals of both biological genesis (Figs. 6–17) and geological genesis, for example, grey granite (Fig. 19) points to many common features in their morphology, in spite of considerable differences in their deformation behavior. These features are: (1) trajectory of a dangerous crack is determined by geometry of applied loading; (2) dangerous crack possesses a broken profile and rough faces; (3) dangerous crack consists of both fully merged and almost merged pore-like cracks.

The model of growth of a dangerous crack in a neck region of a ductile polycrystalline metal is well-known and thoroughly verified and, therefore, it is accepted as the mechanism of ductile crack growth [12,13]. This mechanism can be illustrated by in situ tension experiment of the polycrystalline aluminum in the column of transmission electron microscope (TEM) (see Fig. 20). Initially opaque aluminum thin foil becomes transparent for electron beam in a narrow strip oriented normally to the tensile axis of sample under its stretching in TEM. This strip is the neck region, where extensive dislocation motion is observed on the TEM screen. It is clearly visible on the TEM screen how mobile <110> dislocations leave thin light area of the foil and come to thick dark regions of the sample. There are many pore-like cracks in this thin light strip, which have a trend to merge with one another (see 1st tension in Fig. 20). As a result, dangerous crack appears and propagates by means of pore-like cracks merging (see 2nd tension in Fig 20). It was shown that pore-like cracks grow along low index crystallographic directions, such as <100> and <110>. The dangerous crack obtains broken or zig-zag profile sometimes called dragon teeth that is clearly visible in the case when the plane of a foil is close to low index crystallographic planes (see plane {100} in Fig. 20).

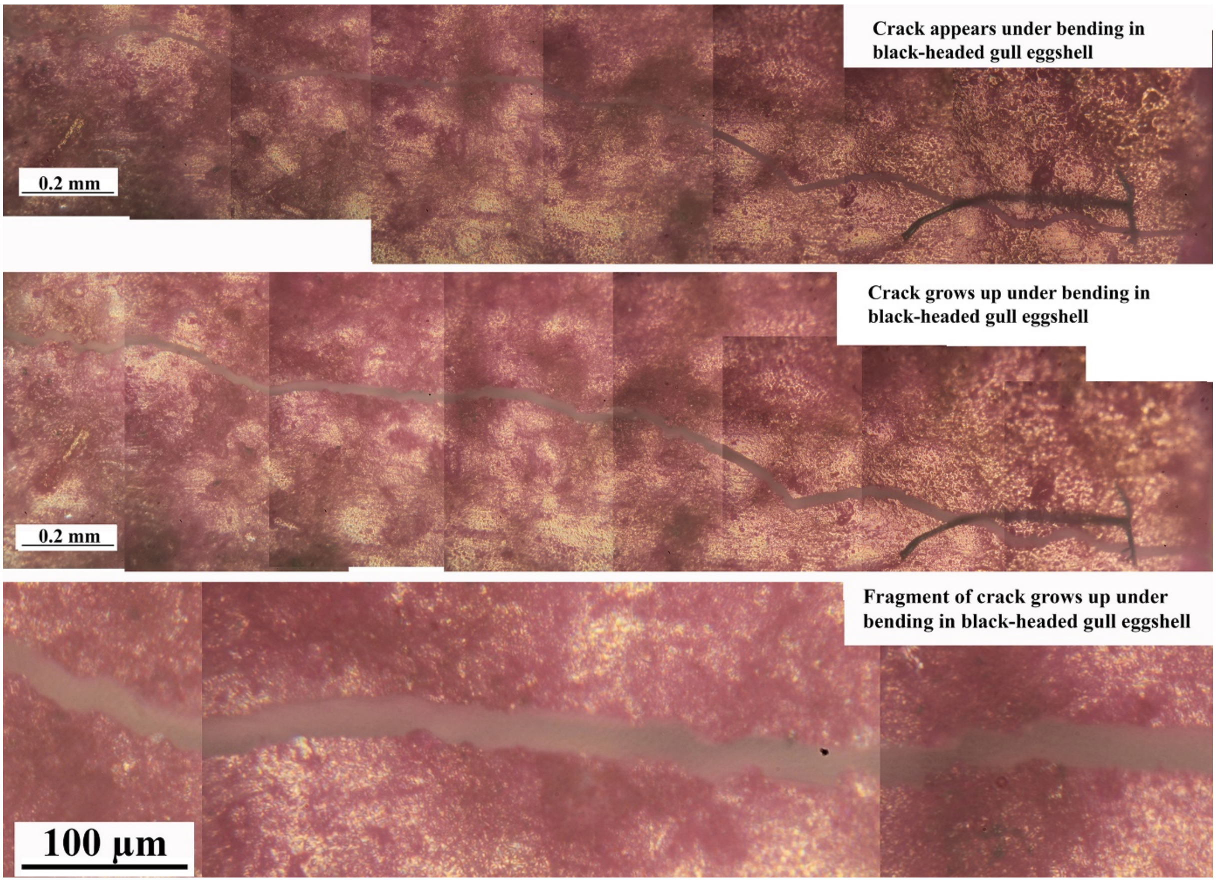

Fig. 6. Evolution of the crack in the sample of black-headed gull eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100 µm).

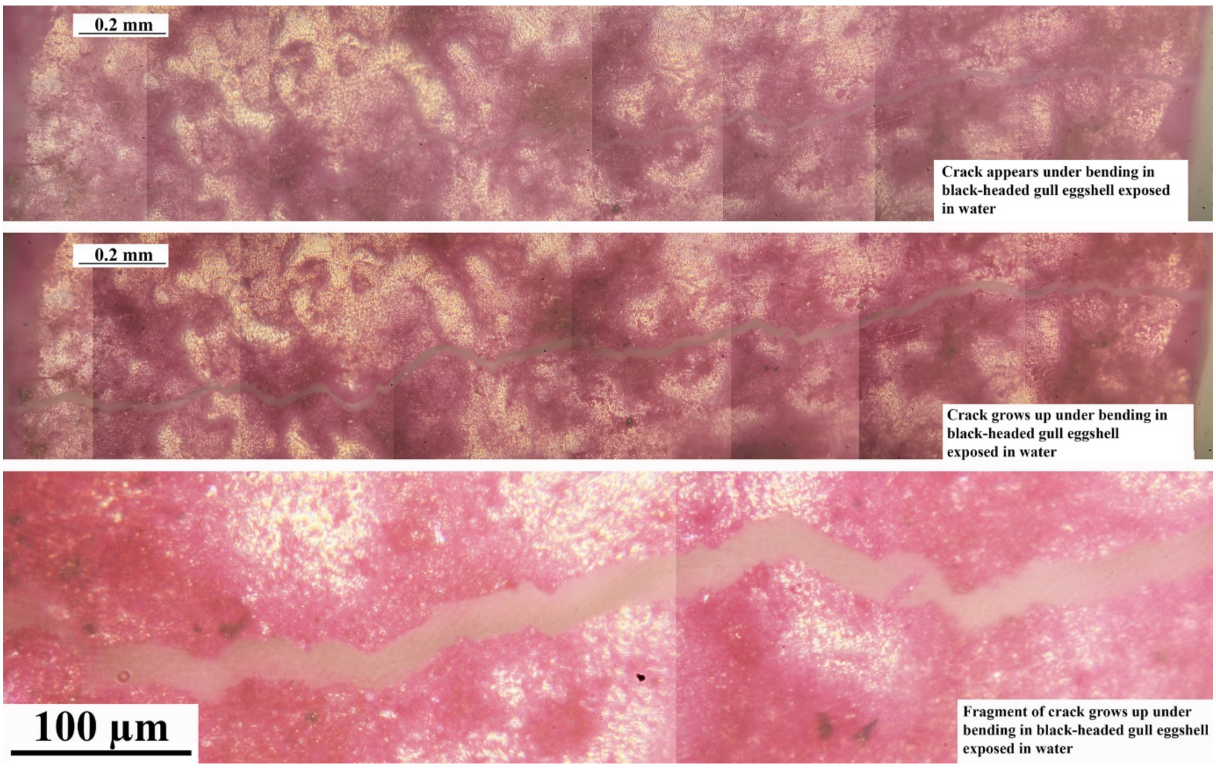

Fig. 7. Evolution of the crack in the sample of black-headed gull eggshell exposed to water under bending: bending 1 (crack appearance); bending 2 (bending deflection is 50 µm).

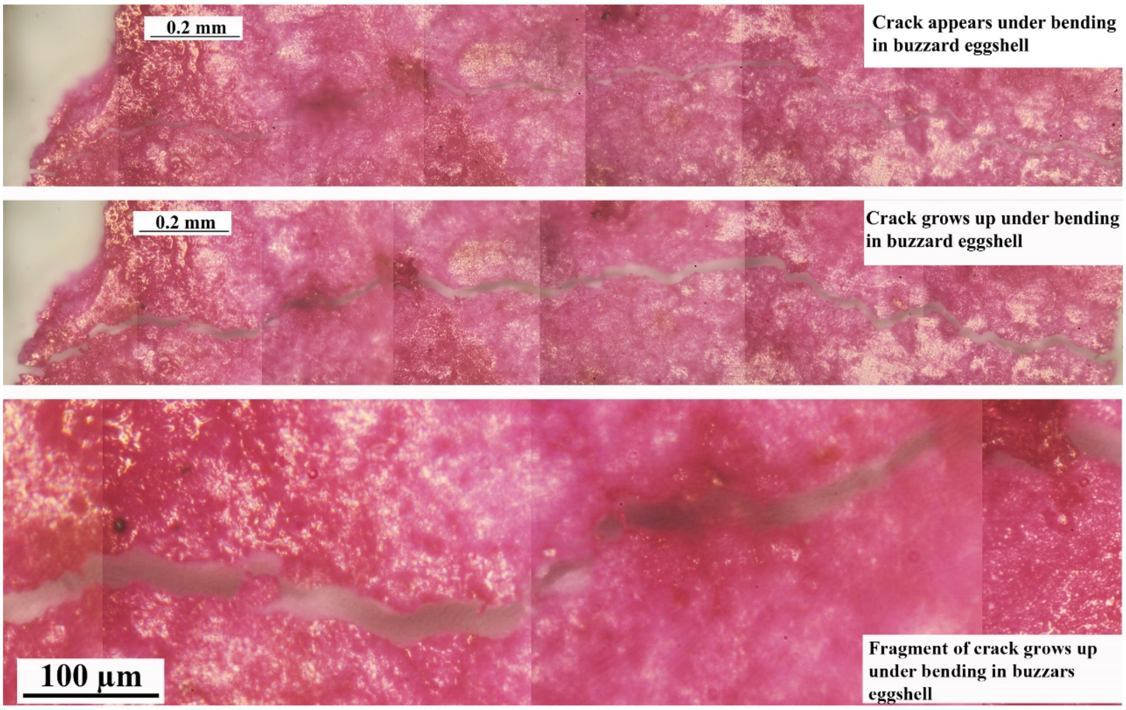

Fig. 8. Evolution of the crack in the sample of buzzard eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100 µm).

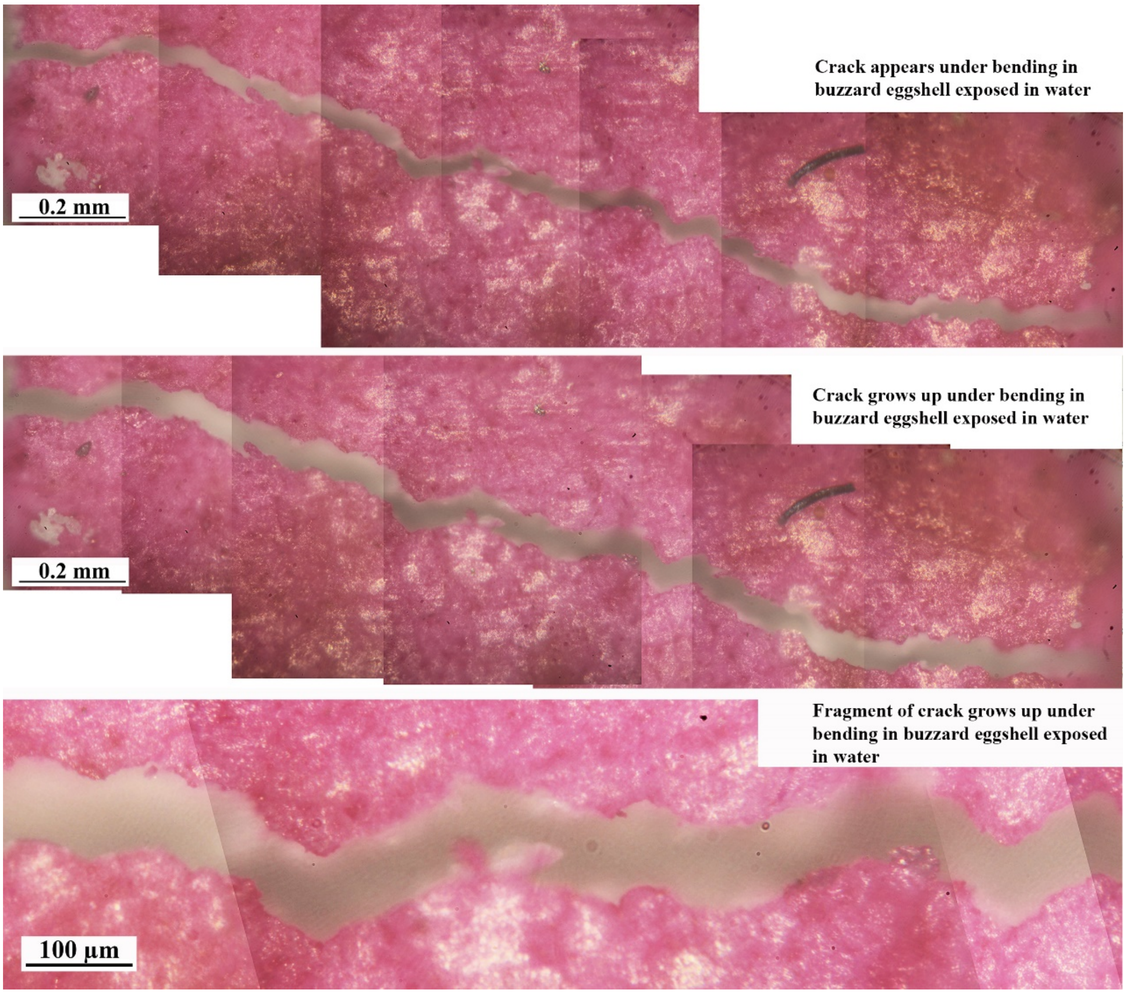

Fig. 9. Evolution of the crack in the sample of buzzard eggshell exposed in water under bending: bending 1 (crack appearance); bending 2 (bending deflection is 50µm).

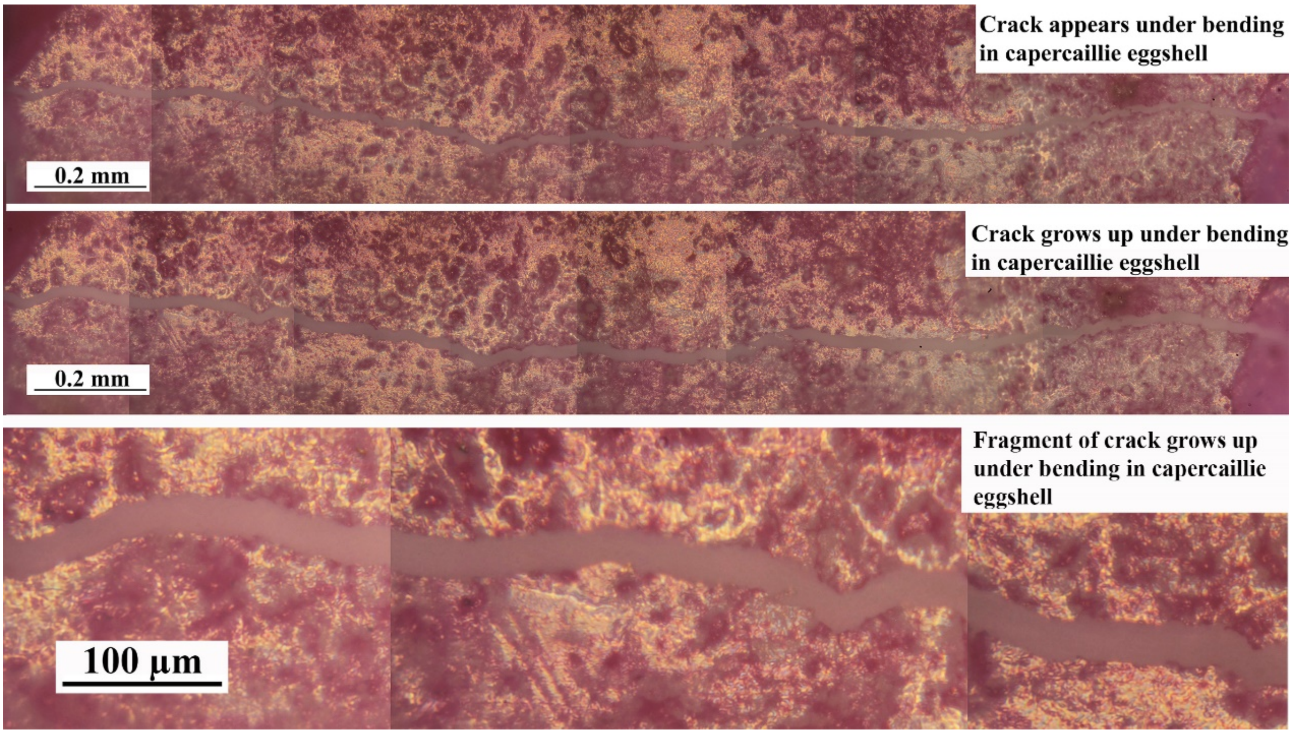

Fig. 10. Evolution of the crack in the sample of capercaillie eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100µm).

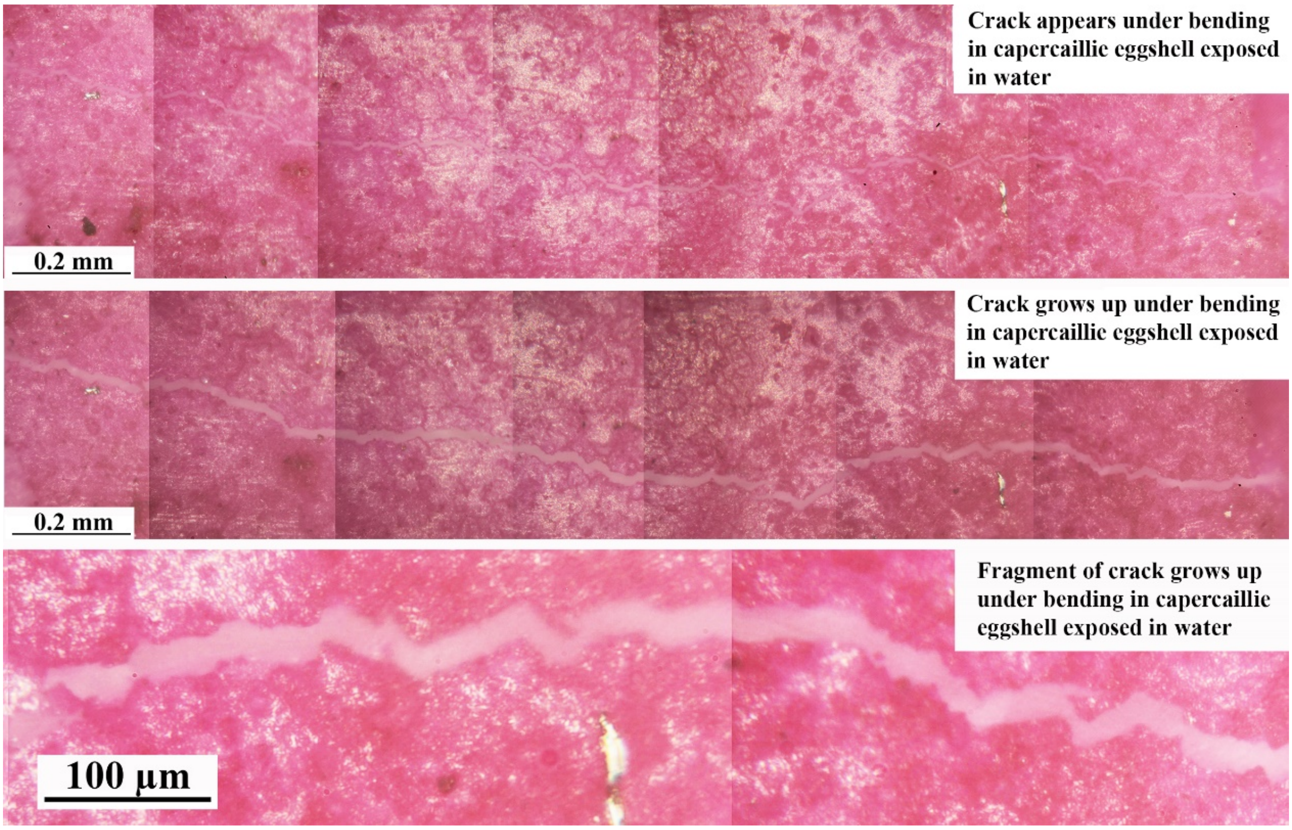

Fig. 11. Evolution of the crack in the sample of capercaillie eggshell under bending in water: bending 1 (crack appearance); bending 2 (bending deflection is 50 µm).

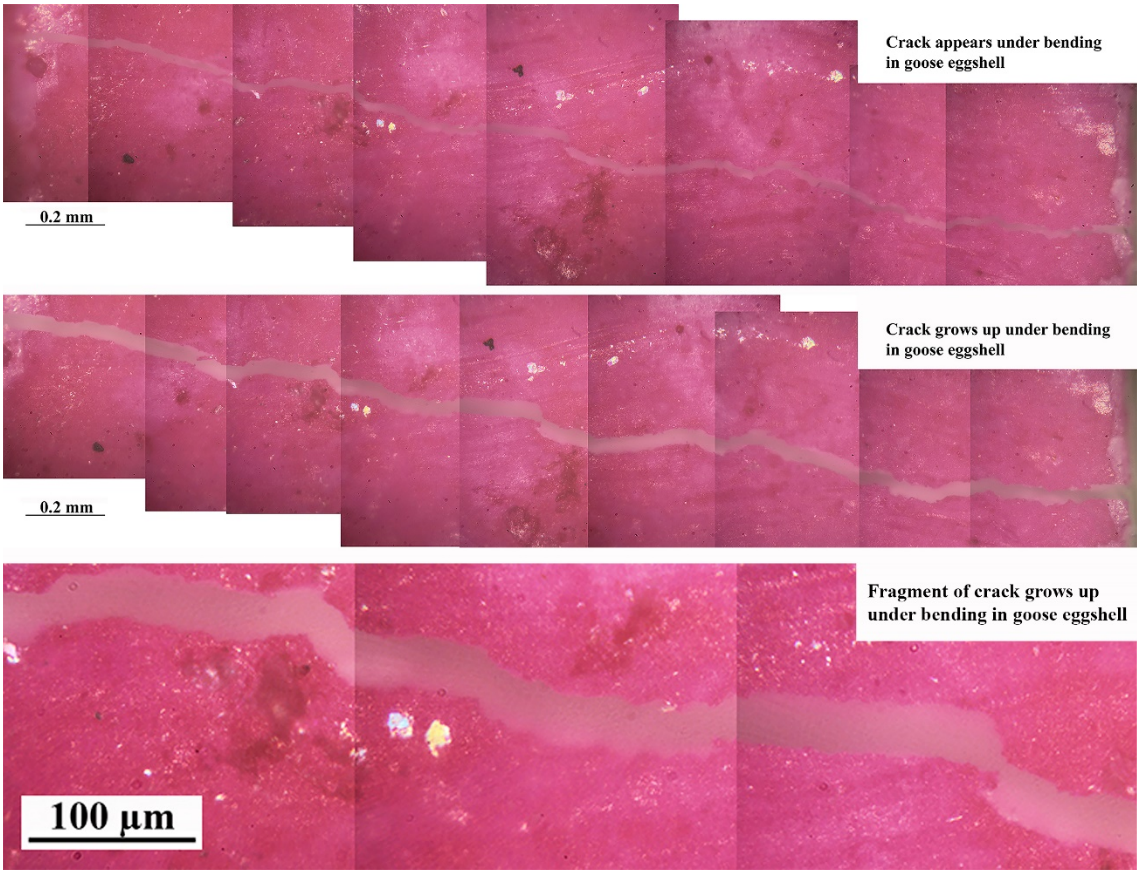

Fig. 12. Evolution of the crack in the sample of goose eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100 µm).

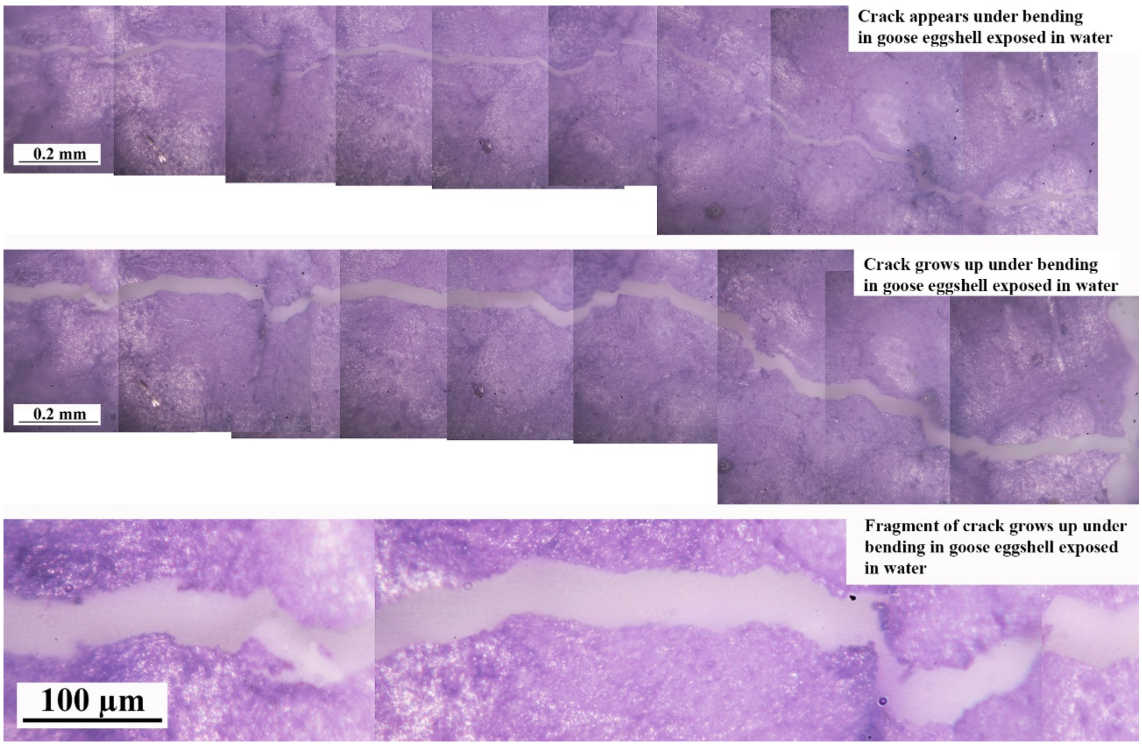

Fig. 13. Evolution of the crack in the sample of goose eggshell under bending in water: bending 1 (crack appearance); bending 2 (bending deflection is 50 µm).

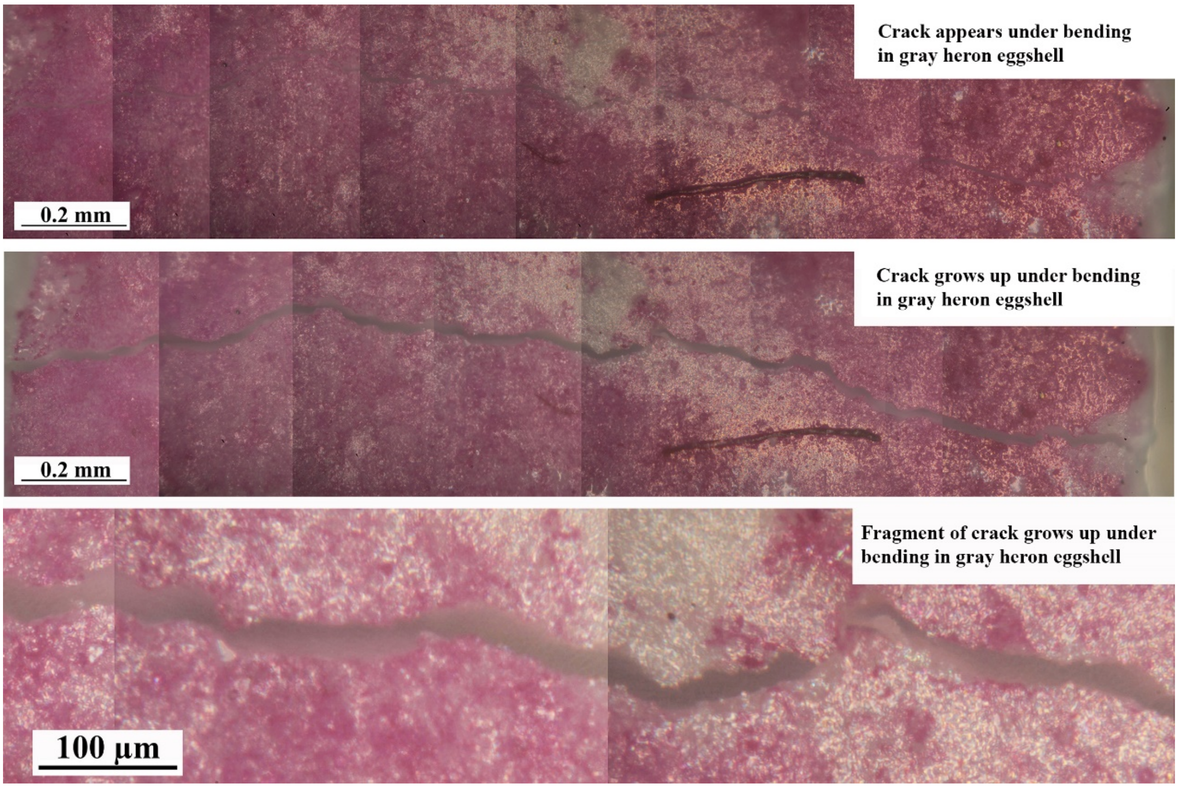

Fig. 14. Evolution of the crack in the sample of gray heron eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100 µm).

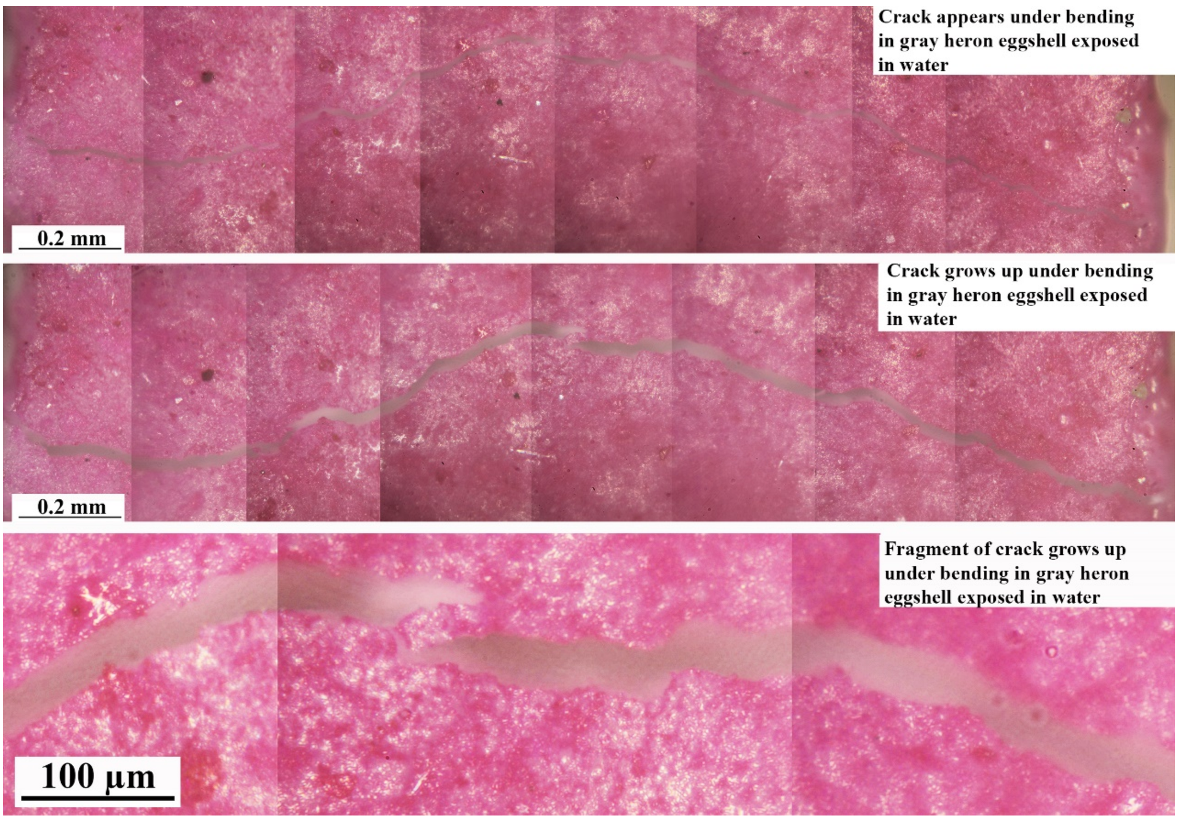

Fig. 15. Evolution of the crack in the sample of gray heron eggshell under bending in water: bending 1 (crack appearance); bending 2 (bending deflection is 50 µm).

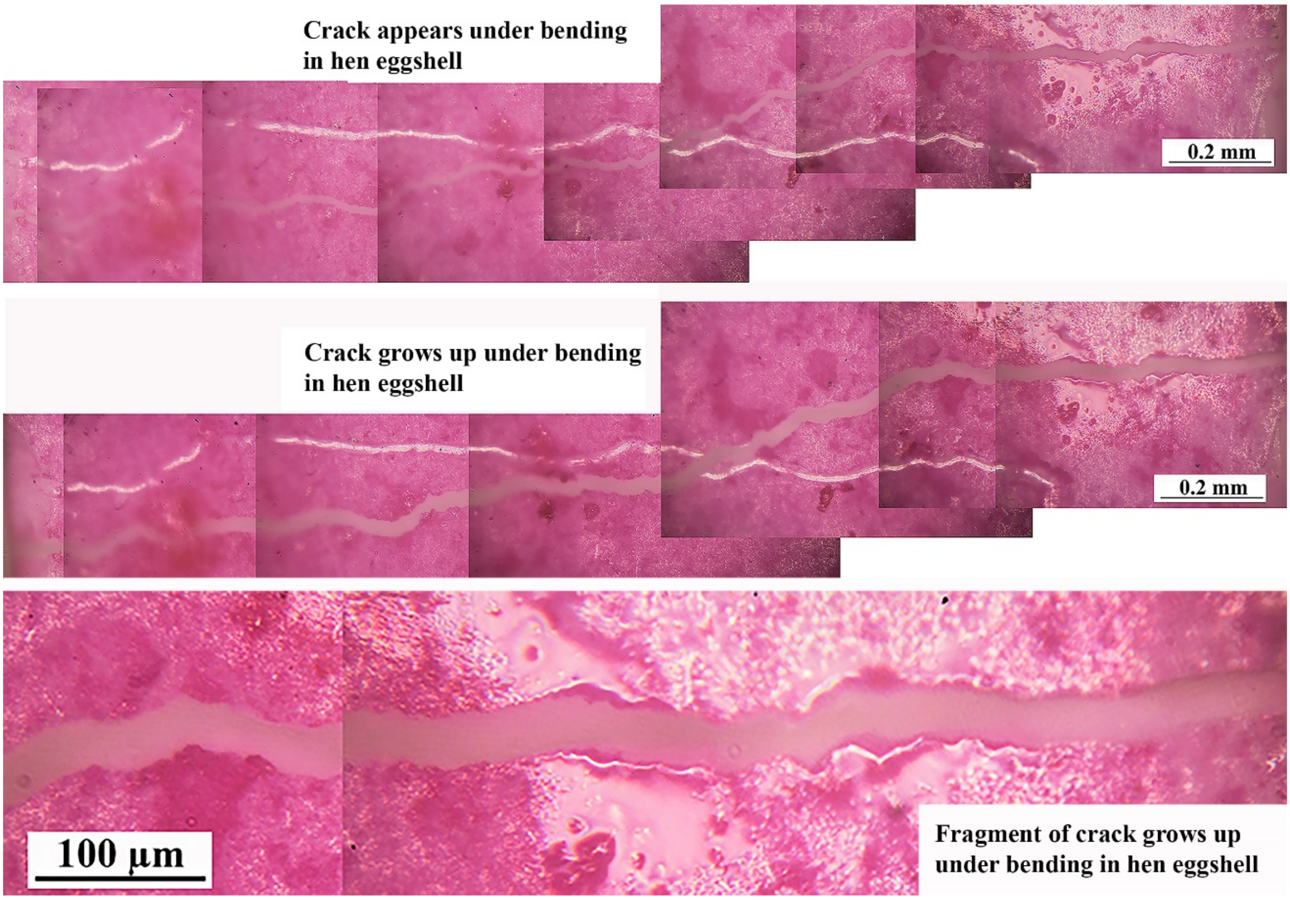

Fig. 16. Evolution of the crack in the sample of hen eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100 µm).

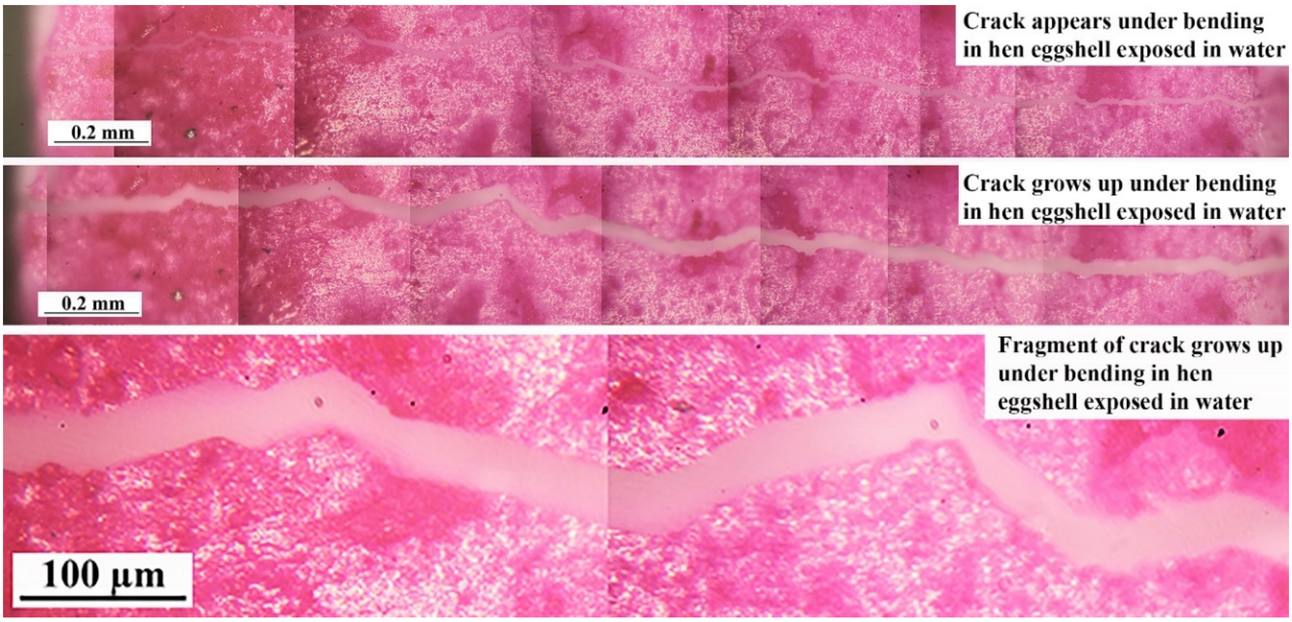

Fig. 17. Evolution of the crack in the sample of hen eggshell under bending in water: bending 1 (crack appearance); bending 2 (bending deflection is 50 µm).

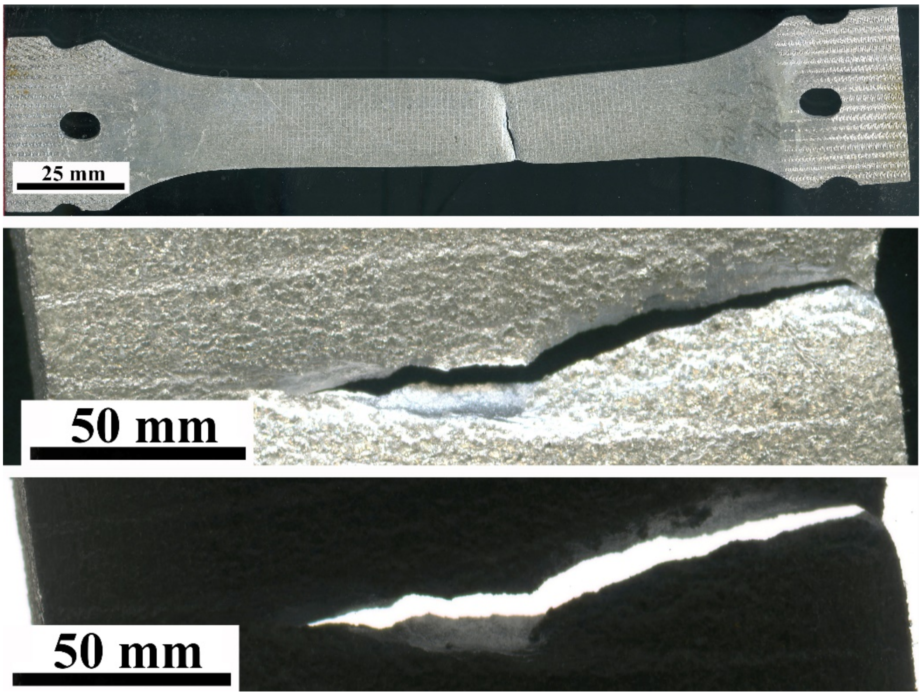

Fig. 18. Crack in the neck of a flat polycrystalline aluminum sample under tension in air.

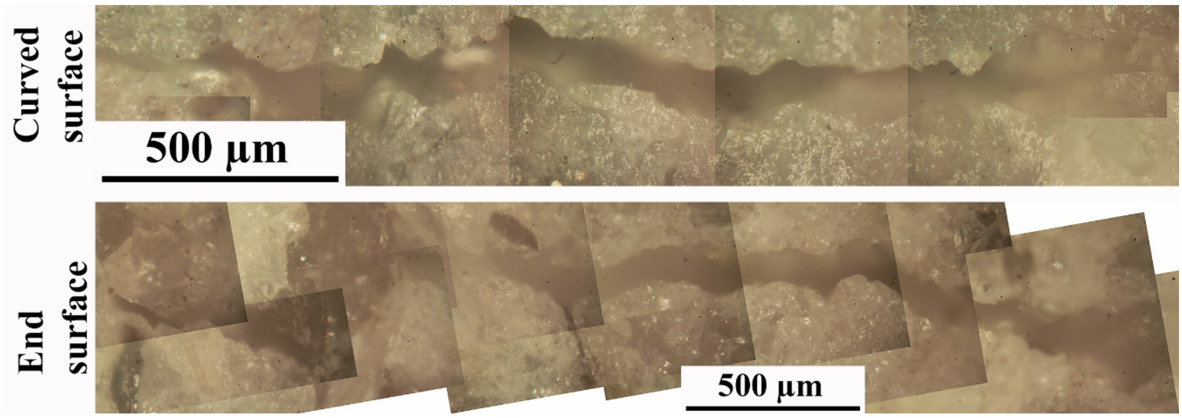

Fig. 19. Crack appears in gray granite under bending in air: (1) fragment of crack that crosses over the curved surface of sample; (2) crack on the end surface of sample, which starts on the edge with the curved surface and ends in its middle part.

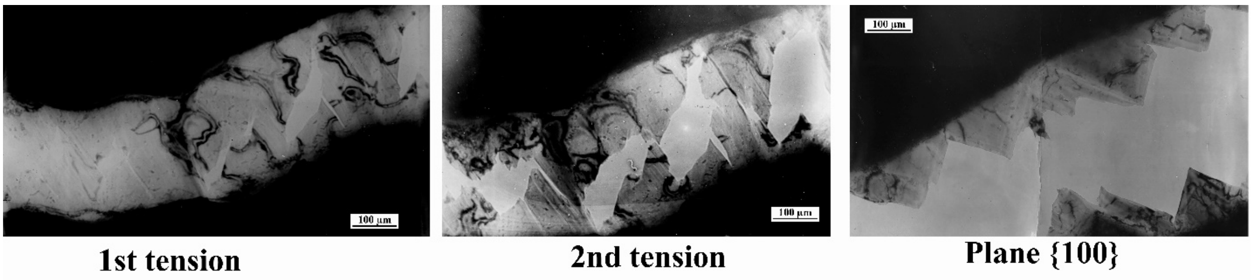

Fig. 20. Crack growth in polycrystalline aluminum thin foil during in situ tension in column of TEM.

It is important to note that no dislocation motion in the thin light region of aluminum foil was observed on the stage when pore-like cracks become merged into the dangerous crack. It means that dangerous crack grows in the material, which is not able anymore to deform plastically because defects-carriers of plastic deformation are absent. This feature may be considered as a property that is inherent to both metals and minerals at the moment of appearance and motion of a dangerous crack. Indeed, a dangerous crack appears in materials, which cannot be plastically deformed because resource of plasticity of metal is almost exhausted in neck region of a metallic sample or a mineral cannot be irreversibly deformed in principle, at least on the macroscopic scale [14,15]. In other words, dangerous crack, whose morphology was described above, appears after considerable plastic deformation in ductile metals, while it nucleates without preliminary irreversible deformation in minerals.

Discussing deformation behavior of a mineral of biological genesis (biomineral) containing bioorganic components, it should be taken into account that bioorganics could influence mechanical properties of its inorganic matrix, particularly ones related to cracking, as it occurs in a tooth enamel [16]. Indeed, deformation prior to failure of samples cut from a bird eggshell under bending is higher than that for samples cut from grey granite or other rocks. The boiling of samples cut from hen eggshell at 100 °C cardinally changes the structure and properties of its bioorganic component. However, this procedure was not reflected neither in deformation behavior on the macroscopic scale nor in crack growth in the boiled samples under bending in comparison with not boiled samples. Mechanical properties of boiled hen eggshell do not change, while crack growth in them is also similar to described above (Fig. 21). It means that mechanical properties of samples cut from hen eggshell do not depend on structural state of bioorganics in hen eggshell and, hence, an influence of bioorganic component on deformation behavior of the samples should be minor. Samples for bending cut from hen eggshell possess two natural surfaces in comparison with samples for bending cut from granite or other rocks of geological genesis. These surfaces could be estimated as almost perfect ones that contain minimal level of stress concentrators. On the contrary, mechanically polished samples of minerals contain a lot of defects that serve as stress concentrators. It seems that it is the dominant cause for the difference between deformation prior to failure under bending for samples cut from hen eggshell and samples cut from rocks.

Findings presented above have clearly shown that dangerous crack in a bird eggshell, has some features inherent to dangerous crack in such ductile metal as aluminum, despite its brittle deformation behavior on the macroscopic scale. In so doing, the fracture mode of a bird eggshell is determined as the brittle transgranular fracture. Is it an artefact or a native response of this biomineral?

Despite advance in understanding of the nature of brittle fracture [17,18], some questions in this sphere continues to be unclear yet. For example, the sole refractory FCC-metal iridium exhibits the brittle transgranular fracture or the transcrystalline cleavage after considerable elongation that can reach few dozens of percents at room temperature [19,20]. The source of transgranular brittle fracture in iridium single crystals under tensile loading, at least on the microscopic

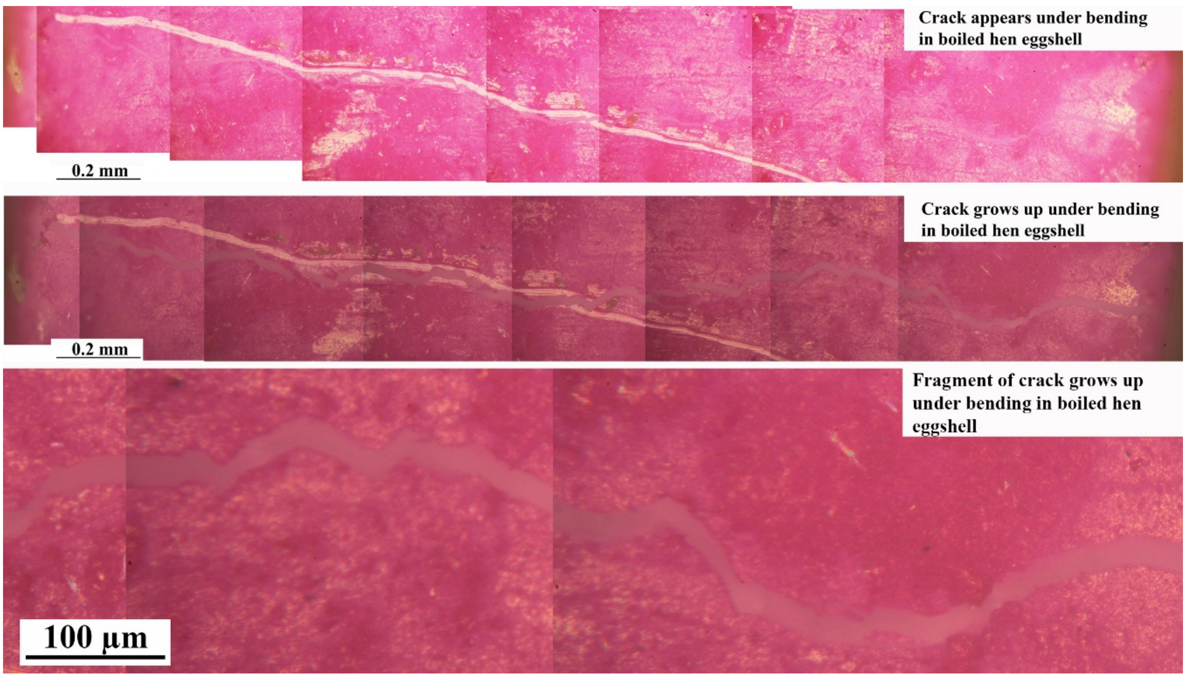

Fig. 21. Evolution of the crack in the sample of boiled hen eggshell under bending in air: bending 1 (crack appearance); bending 2 (bending deflection is 100 µm).

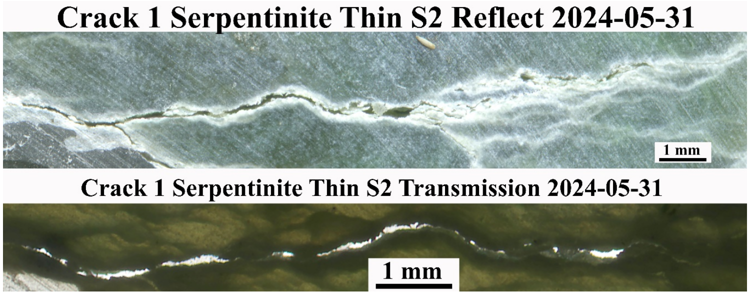

Fig. 22. Fragment of the crack in the thin sample of green serpentinite in the reflected light and in the transmission light.

scale, is V-shape cleavage cracks [21]. It was shown that sometimes these cracks can behave as brittle cracks, while sometimes they behave as a notch in a ductile metal [22]. In the first case, the angle of crack opening is about few degrees as it should be for a crack in an intrinsically brittle solid. In the second case, the angle of crack opening is in the range of 10–15 degrees. In other words, even considerable plasticity of a material is not a-priori an insurmountable barrier for the transcrystalline cleavage.

Hence, the brittle transgranular fracture as fracture mode does not exclude an ability of a solid to exhibit some ductility at least on the microscopic scale even if a material possesses covalent chemical bonding as an eggshell or a rock. Indeed, dangerous cracks in the bird eggshell samples under bending demonstrate the following features of a ductile crack growth: (1) step-by-step growth under step-by-step loading; (2) their morphology features certain similarity to a crack in the neck region in a metal, such as a broken or zig-zag profile.

As it was mentioned above, the plastic zone ahead of dangerous crack, where pore-like cracks appear and merge together, is the important feature of a ductile crack growth [23–25]. Direct observation of the plastic zone in bulk samples of rocks is difficult because of their poor deformability. However, this problem is successfully solved in the thin samples of a rock using an optical microscope operating in the transmission light. Pore-like cracks in the plastic zone in the thin sample of green serpentinite are shown in Fig. 22. There are narrow white areas that look like cuffs around pore-like cracks in the reflected mode images, which are thinned areas of the material near the cracks if the dangerous crack is observed with a help of a light microscope in the transmission mode.

Therefore, it may be supposed that additional channel for stress accommodation becomes active in the samples cut from a bird eggshell under bending. Of cause, the contribution of this channel to the deformation behavior of an eggshell is much less than the contribution of the main channel or the cracking. As a result, deformation behavior of a bird eggshell under bending on the macroscopic scale is governed by the covalent chemical bonding and can be attested as brittle. On the other hand, its influence is large enough that a dangerous crack in both eggshell and rock look like a ductile crack on the microscopic scale. Perhaps, this is the cause why mechanics of cracks as the part of fracture mechanics meets with troubles when applied to some rock materials. Also, it should be pointed out that mechanism of stress accommodation for this additional channel is not attested yet and, hence, it needs detailed study and wide discussion.

5. CONCLUSION

It was shown that deformation behavior on the macroscopic scale of eggshell of such birds as black-headed gull, buzzard, capercaillie, gray heron, goose, and hen under bending is brittle as it should be in material with covalent chemical bonding like minerals and rocks. The fracture surface morphology of the samples agrees with this conclusion. However, the cracking of the samples cut from a bird eggshell under bending has the features, which are inherent to cracking in neck region of the aluminum sample under tension. The first feature is the morphology of the dangerous crack in a bird eggshell which is close to a crack in a neck of a ductile metal. The second feature is the ability of dangerous cracks in a bird eggshell to grow under bending without failure of sample. Hence, a bird eggshell under bending exhibits some features of ductile fracture on the microscopic scale. Also, the Rehbinber's effect for a rock manifests in a bird eggshell under bending in water, when the strength decreases in comparison with testing in air, but the morphology of dangerous crack continues to be the same.

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Об одной особенности деформационного поведения скорлупы птичьих яиц

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Аннотация. Изучено деформационное поведение скорлупы яиц разных птиц при 3-х точечном изгибе. Скорлупа птичьих яиц – это биоминерал, состоящий примерно на 90% из карбоната кальция. В работе была изучена скорлупа яиц нескольких видов птиц: из каждой было вырезано по два набора образцов. Механические испытания проводили на сухих и влажных образцах на воздухе. Трещины в образцах из скорлупы при изгибе изучались in situ на световом микроскопе. У всех видов птиц скорлупа демонстрировала хрупкое поведение в макроскопическом масштабе как в сухом, так и во влажном состоянии. Однако ширину трещины можно увеличить, увеличивая стрелу прогиба, подобно тому, как это происходит в пластичных металлах при растяжении. Вода вызывает некоторое снижение прочности яичной скорлупы при изгибе. Морфология трещин в яичной скорлупе при изгибе оказалась подобной морфологии трещины в области шейки плоского алюминиевого образца. На этом основании делается заключение, что скорлупа птичьих яиц при изгибе в микроскопическом масштабе демонстрирует признаки вязкого разрушения.

Ключевые слова: биоминералы; скорлупа птичьих яиц; изгиб; деформация; разрушение