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NEW APPROACH OF EGGSHELL MECHANICAL PROPERTIES DETERMINATION

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Abstract

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The paper describes a new approach for determination of mechanical properties of hen's eggshell. The suitability and applicability of a Berkovich indentation is discussed. The eggshells were tested in the area surrounding equator line. The deformation modes active during indentation have been examinined from the shape of load-displacement curves. According to measured dependencies, the eggshel shown an viscous-elastic deformation.The values of Young's modulus *E* obtained from radial and tangential directions did not vary significantly. This fact shows on isotropic nature of eggshell structure. It was found that values of *E* do not significantly change neither around the circumference of the equator. The values obtained within this research correspond to values reported in literature and obtained on macroscopic samples. Nanoindentation was found to be a precise and powerful tool, suitable for determining local variations of mechanical properties of eggshells.

egg shell, modulus of elasticity, mechanical properties

Precise determination of eggshell strength properties enables accurate description of eggshell mechanical behaviour, which is important e.g. for design of egg-processing and transporting equipment. Material strength concerns the strength of the main elements of a material and is described namely by the elastic modulus E. There are several methods to determine the eggshell's modulus of elasticity. Some of them determine the static modulus of elasticity E_s (Lin et al., 1993; Dhanoa et al., 1996; Buchar et al., 2001), other measure the dynamic modulus of elasticity E_d (De Ketelaere et al., 2002; Kemps et al., 2004, Lin et al., 2004). It was proven that eggshell strength depends on many factors such as diet (De Andrade et al., 1977; Grizzle et al., 1992; Lichovníková et al., 2008), breeding conditions (Lichovníková and Zeman, 2008), hen breed (Máchal and Simeonovová, 2002; Máchal et al., 2004), egg shape (Havlíček et al., 2008; Nedomová et al., 2009) and other parameters.

Knowledge of mechanical properties at microscale level would considerably improve the quality of mechanical behaviour prediction or e.g. precise model creating. But accurate measuring of the local mechanical properties can often be challenging. Assessing e.g. fracture toughness by making direct measurements of cracks created using a sharp diamond indenter, such as Vickers, Knoop, Berkovich, or cube corner, can appear to be an attractive alternative to more traditional testing techniques (Lawn et al., 1980; Anstis et al., 1981; Fett, 2002; Fett et al., 2005). These techniques are used with success in studying the mechanical characteristics of biomaterials and hard tissues (Khor et al., 2003; Denry and Holloway, 2004; Imbeni et al., 2005; Mullins et al., 2007). Indentation techniques for assessing micromechanical properties are attractive due to the simplicity and expediency of experiments, and because they potentially allow the characterization of both local and bulk fracture properties. Generally speaking, there are four main indentation techniques: the Vickers indentation fracture (VIF) test, the cube corner indentation fracture (CCIF) test, the Vickers crack opening displacement (VCOD) test and the interface indentation fracture (IIF) test. In general, the VIF and CCIF techniques are found to be poor for quantitatively evaluating toughness of any brittle material, and the large errors involved (approx. ±50%) make their applicability as comparative techniques limited (Kruzic et al., 2009).

Three factors motivate the use of nanoindentation – depthsensing indentation testing at very small length scales, realistically ranging from nanometer to micrometer lengthscales – for the study of the mechanical properties of natural materials. First, in such tests the load and displacement of a small probe, the indenter tip, are continuously monitored as the probe is loaded onto the surface of interest, rendering the method ideal for probing local gradients and heterogeneities in natural materials and for examining their hierarchical and multiscale organization. Second, a major factor in the recent rapid growth in nanoindentation is that no extensive sample preparation is required prior to mechanical testing, in sharp contrast to techniques such as tensile testing for which samples must be cut or machined. Third, most nanoindentation instruments provide experimental control that allows for purposeful exploration of a variety of different deformation modes by changing experimental time scale, indenter tip geometry, and loading conditions (Oyen and Cook, 2009).

This paper presents some new experimental results on using Berkovich indentation to assess the elasticity modulus of hens eggshell. The method is based on the direct measurement of the load-displacement relationship using a very small tip pressed into the material surface. The depth of penetration starts from the nanometer scale. Received values are compared with values obtained from traditional experiments.

MATERIALS AND METHODS

Eggshells

Eggs (*Hisex Brown* strain) were used for the experiment. Hens were kept in cage technology in a commercial breeding farm in the Czech Republic. Eggs were collected in the hens age of 75 weeks. Deffective eggs were not included in the experiment.

Preparation of specimens

The chips of eggshells (originating from five different positions around egg equator) were embedded into metacrylate tablet. In order not to thermally affect the structure, the specimens were cold-prepared. Commercially available two-component resin was used for metacrylate mixture preparation and the specimens were left to dry and cure for 8 hours. The tablets were polished in order to achieve flat surface with maximum roughness of 10–20 nm.

Experimental set-up an loading conditions

Nanohardness tester CSM was used to perform the experiments. Standard Berkovich tip was brought to the sample surface producing a series of imprints. The indenter has a nominal tip radius of $r < 20$ nm and an inclined angle of $\theta = 65.3°$.

Load versus depth of penetration was measured through the whole procedure of loading, holding, and unloading. Loading and holding parts of the diagram contain elastic, plastic and viscous deformations, whereas the unloading part is usually supposed to be elastic. Elastic constants are extracted from this part using semi-analytical elastic solutions (Oliver et al., 1992).

The eggshell chips were loaded in both radial and tangential directions. Trapezoidal loading diagram with linear loading, unloading and intermediate holding period was performed.

RESULTS AND DUSCUSSION

Mechanical properties of biological materials are increasingly explored via nanoindentation testing. Since the mechanical properties of hen's eggshell are strongly affected by many factors, single variable evaluation must be considered carefully and with respect to complex character of given material.

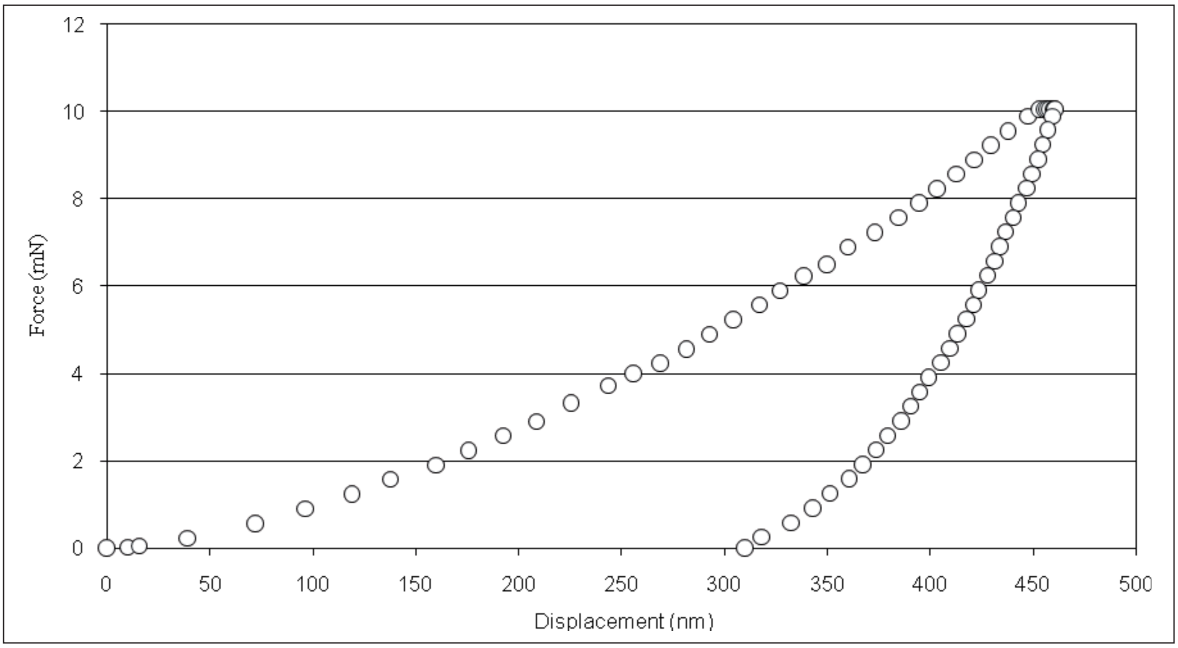
The avian eggshell is a highly ordered calcitic bioceramic composite, with both inorganic and organic constituents. The interactions between the inorganic and organic components within the structure are poorly understood but are likely to occur at the nanometre level. Thus structural variation at this level may impinge on the overall structural integrity and mechanical performance of the eggshell, and therefore analysis at this level is fundamental in fully understanding this ordered structure.

The series of nanoindention experiments (in two loading directions described above) has been performed with following parameters: peak load = 10.00 mN, loading rate = 20.00 mN/min, unloading rate = 20.00 mN/min, holding period = 10.0 s and following results: Young's modulus (radial loading) = 41 ± 3.9 GPa, Young's modulus (tangential loading) = 43 ± 4.1 GPa. The calculated and values represent 5 individual measurements performed on the specimens originating from 5 different position around the equator. The value of Poison's ratio ν (0.345) has been adopted from measurement described in Nedomová et al. (2009), where the similar eggshels were used (the research was conducted in parallel with here presented one and the same eggs were used). Other authors report similar values of ν, e.g. 0.307 (Lin et al., 2004) or 0.300 (Couke, 1998), but in these cases, different eggs (hen strain, age etc.) were used.

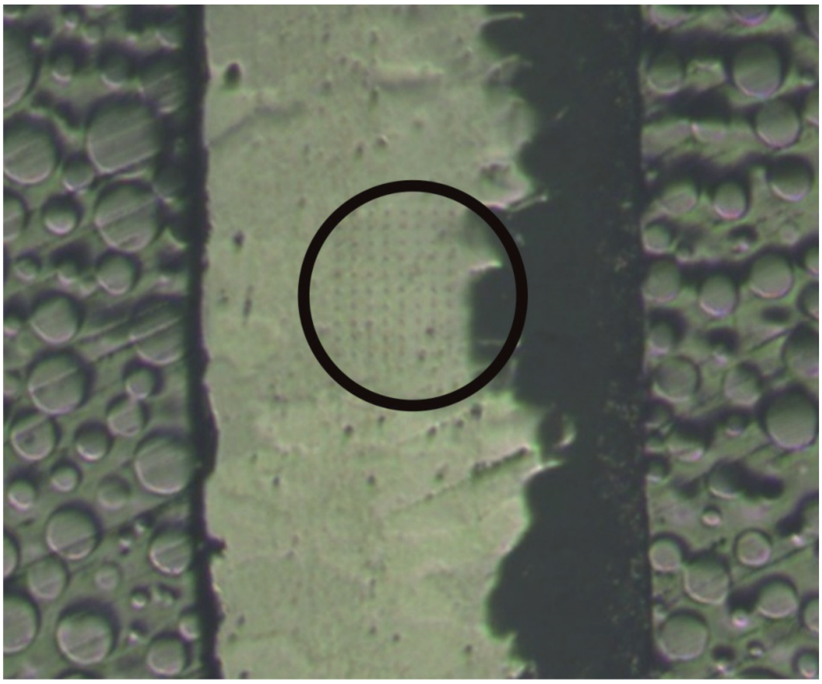
The example of loading diagram is shown in Fig. 1.

The values of Young's modulus E are in general agreement with values reported by Nedomová et al. (2009), where the experiments have been performed using similar eggshells. Nedomová et al. (2009) found that elastic constants are independent on the egg shape as well as loading force orientation (egg loaded either on equator strip and/or egg-tip). This conclusion was supported by numerical simulations.

The values of E obtained from radial and tangential directions did not vary significantly (41 ± 3.9 GPa and 43 ± 4.1 GPa, respectively). This scatter shows on isotropic nature of the eggshell structure. Due to small number of tests, this conclusion has to be confirmed by further and more detailed experiments, but it already shows on the general trend. The values



1: *Load versus depth plot*



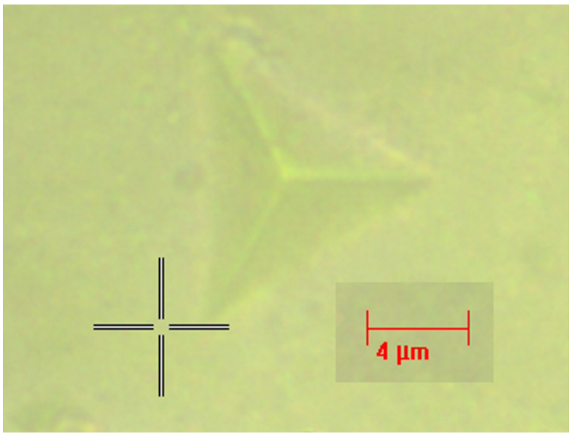
2: *Eggshell cross section*

calculated for measurements performed around the equator did not change significantly, thus the micro mechanical properties seem to be independent on the position around the eggshell circumference. The edges, membrane, inhomogeneities and pores were avoided in the measurement. The overall view (100x magnification) on the eggshell cross section is shown in Fig. 2. The beads or little circles on the picture margins represent undissolved particles of metacrylate. Area indicated by the black circle in the middle of the picture shows the matrix of 80 indents (10 x 8), which the average values for given position were calculated from. The left edge of the eggshell (as shown in the Fig. 2) is the outer side while right edge is the inner side with membrane.

The surface structure appears to be fairly homogenous, which supports the micromechanical measurements.

Fig. 3 shows a detailed view on the cross sectional surface with an indent.

It follows from macroscopic measurements that mechanical properties show certain degree of va-

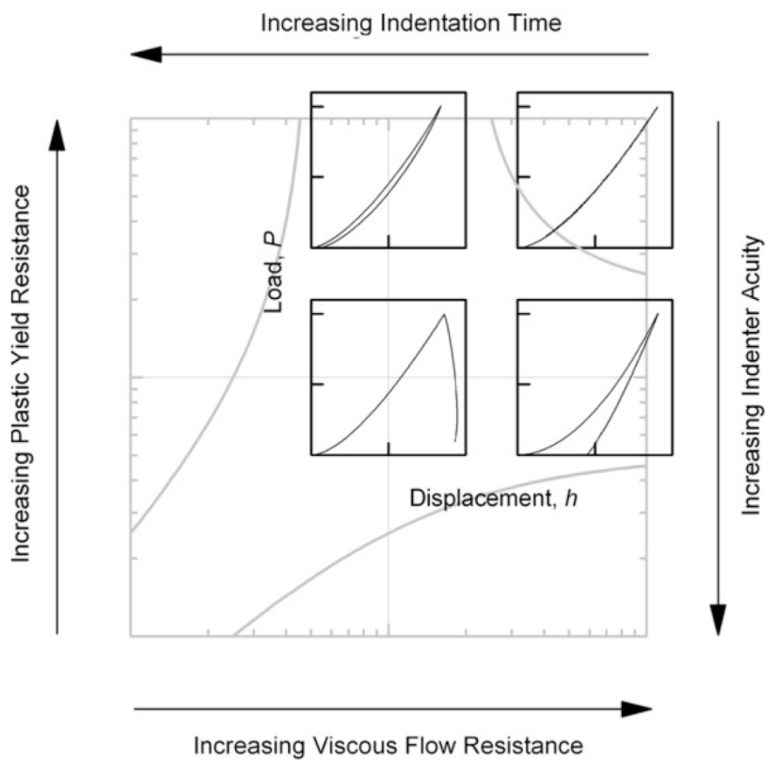


3: *Indentation imprint (load applied = 10 mN)*

riability when measured at different locations on the same egg (Lin et al., 1995). Therefore, the use of nanoindentation for creating of a more detailed „map" of eggshell's micromechanical properties from various locations seem to be a promising and probably one of very few applicable approaches.

Oyen and Cook (2009) presented a scheme for ascertaining which deformation modes are active during a particular indentation test based on the load-displacement trace. They presented two behavior maps for indentation, one in the viscous-elastic-plastic space, concerning homogeneous deformation, and one in the plastic versus brittle space, concerning the transition to fracture behavior when the threshold for cracking is exceeded. It is reasonably straightforward to identify the deformation modes active during indentation testing simply by examining the shape of the indentation load-displacement. According to measured dependencies, the eggshel shown an viscous-elastic deformation. There is a continuum of responses with different relative amounts of viscous, elastic and plastic deformation, and these possibilities can be illustrated compactly on a deformation mechanisms map. Example of such map is shown in Fig. 4. (reworked version of a picture presented in Oyen and Cook, 2009). Construction of an eggshell map will be presented in our following paper based on larger volume of input data.

Presented results approved that nanoindentation, as a novel testing method used for determining of eggshell micromechanical properties, offers a precise tool for describing material characteristics on eggshell micro-scale and thus offers precise and valuable data for mapping of material parameters, modeling, numerical simulations and procedures used for upgrading of the current state of knowledge at this particular field. In all of the specific cases of biological materials including eggshells, a richness of information is found in the indentation response when an analysis method is selected appropriate for the modes of deformation actually present in the tissue under contact probe loading.



4: *Indentation behavior map overlaid for (clockwise from upper right-hand corner) elastic (E), elastic-plastic (EP), viscous-elastic-plastic (VEP) and viscous-elastic (VE) material responses*

SUMMARY

The objective of the present study was to investigate the potential utilization of nanoindentation as a tool for determination of eggshell's micro-mechanical properties and to compare the experimental results with results obtained from conventional tests.
Eggs (*Hisex Brown* strain) were collected from a commercial packing station in the Czech Republic. The eggshell chips from equatorial position on the egg were embedded into metacrylate tablet. The tablets were dried and polished in order to achieve flat surface with maximum roughness of 10–20 nm.
Nanohardness tester CSM was used to perform the experiments. Load versus depth of penetration was measured through the whole procedure of loading, holding, and unloading. Elastic constants are extracted from this part using semi-analytical elastic solutions.
The eggshell chips were loaded in both radial and tangential directions. Trapezoidal loading with linear loading, unloading and intermediate holding period was performed.
The values of Young's modulus *E* are in general agreement with values reported by Nedomová et al. (2009), where the experiments have been performed using similar eggshells.
The values of *E* obtained from radial and tangential directions did not vary significantly (41 ± 3.9 GPa and 43 ± 4.1 GPa, respectively). This scatter indicates isotropic nature of the eggshell structure. Also the values for measurements performed around the equator circumference did not change significantly.
It has been approved that Berkovich indentation offers relevant and precise tool for describing material characteristics on micro-scale level. Obtained data can be consequently used e.g. for modeling or numerical simulations of eggshell mechanical behavior.

SOUHRN

Nová metoda stanovení mikromechanických vlastností vaječné skořápky

Předkládaná práce popisuje metodu nanoindentace a hodnotí její možnosti při stanovení lokálních mikromechanických vlastností skořápek slepičích vajec. Práce též srovnává získané experimentální výsledky s výsledky tradičních testů.
Testována byla vejce slepic plemene *Hisex Brown*. Vzorky skořápek odebrané ze střední části vejce (rovníku) byly zality do dentakrylových tablet. Tablety byly následně vysušeny a vyleštěny tak, aby drsnost povrchu nepřesahovala hodnotu 10–20 nm.
Experimenty byly provedeny na nanoindentačním zařízení CSM. Během zatěžování, zádrže i odtěžování byla sledována závislost hloubky indentace na síle. Pomocí analytických řešení byly z těchto závislostí vypočteny elastické konstanty. Vzorky skořápek byly zatěžovány v radiálním a tangenciálním směru.
Hodnoty *E* získané při zatěžování v radiálním a tangenciálním směru se nelišily statisticky významným způsobem (41 ± 3.9 GPa a 43 ± 4.1 GPa). Tento výsledek naznačuje isotropní charakter struktury vaječné skořápky. Významně se nelišily ani hodnoty naměřené po obvodu rovníku. Hodnoty *E* se v zásadě shodovaly s hodnotami uváděnými Nedomovou et al. (2009), kde byl *E* hodnocen jiným postupem na obdobných vzorcích.
Bylo prokázáno, že nanoindentace představuje relevantní a přesný nástroj pro stanovení materiálových charakteristik vaječných skořápek na mikroskopické úrovni. Získaná data mohou být dále využita např. pro tvorbu modelů nebo numerické simulace chování vajec.

vaječná skořápka, modul pružnosti, mechanické vlastnosti

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