COMPOSITION, STRUCTURAL AND MATERIAL PROPERTIES OF LEECH TEETH – EXAMPLE OF BIOINSPIRATION IN MATERIALS RESEARCH

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Keywords: bioinspiration, atomic spectroscopy, nanoindentation

1. Introduction
The leech's sucking apparatus is an amazing instrument – it has 3 jaws and 300 teeth made for easily cut into the skin of the host animal. In ancient India and Greece, leeches have been used in medicine to remove blood from patients. Today, leeching is used rarely and the use of leeches has shifted into reconstructive and plastic surgery. Although there are a number of papers dealing with the leech stretch receptors, body wall muscles1 or central nervous system CNS2, there is no paper on the composition or material properties of its teeth. In this study we used nanoindentation and atomic spectroscopy to reveal composition and material properties of leech teeth and to demonstrate the optimization possibilities of nature to manufacture these very sharp and tiny blades which can easily penetrate the host's skin.

2. Experimental details
2.1. Sample extraction and preparation
Five samples of leech's jaws were obtained from adult subjects of Hemopis sanguisuga. The subjects were euthanised with ether and sliced in area of sucking apparatus. Individual jaws with length around 500 µm (depicted in Fig. 1) were carefully separated under magnification glass (5×) using a sharp-tip scalpel, micror retractores and pair of tweezers. The samples were cleaned from the soft tissues and embedded in low shrinkage epoxy resin.

The surface of the samples was grinded and polished. Diamond grinding discs followed by monocrystalline diamond suspension were used for grinding procedure3. The best reached final surface roughness average (R a) was 16 nm. All samples were prepared with roughness less than 40 nm, which is adequate value for micromechanical testing.

2.2. Mechanical testing
Quasi-static nanoindentation was performed using the nanomechanical instrument Hysitron TI 950 TribolIndenter™. Berkovich diamond tip, (triangular pyramids with angle of 142.3°) was used to obtain elastic properties of the teeth. The test was performed in three segments. Loading, constant force, unloading phase of the test were prescribed. Maximum force was reached at 5 s, then 2 s of dwell and 5 s of unloading followed.

The first set of 5 indents were performed on the sample with roughness approximately R a = 40 nm. To reduce the influence of roughness on results, a force of P max = 8000–8300 µN was applied resulting in indents depth app. 500 nm. Then the sample was polished again to...
decrease its roughness down to \( R_a = 16 \) nm. The force of \( P_{\text{max}} = 900 \mu\text{N} \) corresponding to \( h_{\text{max}} = 150 \) nm was used at this time. The force-depth curves were plotted for each indent, and reduced moduli were calculated using the Oliver-Pharr method\(^4\).

2.3. Composition analysis

Morphological investigation of leech's teeth depicting its true size and shape has been accompanied with composition microanalysis. The microanalysis was carried out by Bruker Quantax energy dispersive spectrometer installed in Tescan MIRA II scanning electron microscope (SEM). Concentration of individual elements was determined from the relative intensity of their characteristic X-ray spectra by the Esprit program provided by microanalyser manufacturer. By the nature of elemental microanalysis it is impossible to identify molecular composition of the studied matter, only elemental composition is as the result available. From the composition analysis as the significant elements and their respective concentrations are calcium, oxygen and carbon were identified.

3. Results

In this preliminary analysis, the main constituents of leech's teeth have been identified. The tooth is composed mainly of calcium (41.9 %), oxygen (41.2 %) and carbon (11.4 %), other constituents are present in small quantities (F 2.1 %, Na 1.0 %, P 0.9 %, S 0.6 % and Mg 0.6 %). Therefore, a substance typical for mineral component in bones, hydroxyapatite is likely present in the teeth among other substances.

The mechanical properties of two leech teeth were measured in cross-section (depicted in Fig. 2). The average value of reduced modulus \( E_r = 29.41 \pm 1.10 \) GPa was obtained from 5 indents of the first test \( (h_{\text{max}} \approx 500 \) nm, \( R_a = 40 \) nm). In the next measurement of 10 indents, the average modulus was \( E_r = 27.02 \pm 4.03 \) GPa \( (h_{\text{max}} \approx 150 \) nm, \( R_a = 16 \) nm). These values correspond to the values from measurements on cortical bone and tooth dentin\(^5\).

4. Conclusions

From the results of the nanoindentation it could be concluded that the mechanical properties of leech's tooth are independent on indentation depth. High-precision surface preparation allows indenting in small depths with a high accuracy. Another advantage of an achievement of very low roughness can be seen in the possibility for the placement of more indents in the same area. Reduction of indent size is very desirable because of the tiny cross section of the tooth surface, which is smaller than 1000 \( \mu\text{m}^2 \).

Mechanical properties and composition microanalysis of the teeth corresponding to biomaterials such as cortical bone or dentin\(^7\) offers the assumption that it is possible that the tooth surface could be composed of enamel, as it is at higher animal species. Therefore, it would be beneficial to prepare longitudinal cuts of leech's teeth, which would enable the indentation of superficial layer and inner part of the teeth. Assessment of mechanical properties from different anatomical parts could help to determine whether the outer layer is created by enamel, and whether the inner part is composed of dentin. Based on this information, an accurate constitutive material model can be created.

The research has been supported by the Grant Agency of the Czech Republic (grant No. P105/10/2305), Ministry of Education of the Czech Republic: Transdisciplinary research in Biomedical Engineering II. No. MSM 6840770012 and research plan of the Academy of Sciences of the Czech Republic AV0Z20710524.

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J. Šepitka\(^a\), J. Lukeš\(^b\), O. Jiroušek\(^b\), D. Kytýř\(^b\), and J. Valach\(^b\) \(^a\) Czech Technical University in Prague, Faculty of Mechanical Engineering, Prague, \(^b\) Institute of Theoretical and Applied Mechanics, Academy of Sciences of the Czech Republic, v.v.i., Prague, Czech Republic: Composition, Structural and Material Properties of Leech Teeth – Example of Bioinspiration in Materials Research

The leech's sucking apparatus is an amazing instrument – it has 3 jaws and 300 teeth made for easily cut into the skin of the host animal. In this study, we used nanoindentation and atomic spectroscopy to reveal composition and material properties of leech teeth. Five samples of leech jaws obtained from adult subjects of *Hemopis sanguisuga* were investigated. Main constituents of leech teeth have been identified. The tooth is composed mainly of calcium (41.9 %), oxygen (41.2 %) and carbon (11.4 %), other constituents are present in small quantities (F, Na, P and S), and therefore, a substance typical for mineral component in bones hydroxyapatite is likely present in the teeth among other substances. Material properties, which are independent on indentation depth, examined by nanoindentation provided average reduced modulus \( E_r = 27.54 \pm 3.71 \) GPa.
NANOINDENTATION BASED MICROANALYSIS OF HENS' BONES

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Keywords: hen bone, micro-mechanical properties, nanoindentation, elastic constants

1. Introduction

Bone mechanical properties and the presence of fractures in laying hens are both a welfare and an economic concern for the poultry industry. The mechanical properties of the bone have significant importance, especially in understanding fracture behavior as a function of mineralization. If we can obtain an insight into the determinants of the bone strength, then better methods to monitor and select animals with abnormal bones can be identified. The composition of the bone tissue is extremely complex compared to most engineering composites. The organization of the bone within a Haversian system consists of a central canal surrounded by concentric lamella. Lamellae are observed at the level of a light microscope. A number of attempts have been made to describe the biomechanical properties of the bone at this level, but this description does not go beyond the histological level. The mechanical properties of cortical bones (including some micromechanical aspects) were described and reviewed in classical works1. The organization of the bone tissue properties in healthy bones with those of diseased and genetically modified small animal models (including mice, rats, and zebrafish)12.

Nanoindentation has been previously used to compare the bone tissue properties in healthy bones with those of diseased and genetically modified small animal models (including mice, rats, and zebrafish)12. In nanoindentation, a small probe with nano-meter dimensions contacts a flat, prepared surface of a material. The resulting force and contact depth (i.e., displacement) data enable the calculation of elastic, plastic, and viscous material properties13–15 of biological tissues like cortical or trabecular bones16 or eggshells17 at a spatial resolution similar to that of the tissue-level structural features in the bone. Nanoindentation can be also used to measure the creep behavior of biological tissues by fitting the depth vs. time data at constant load to rheological models18. Viscoelasticity may affect both the elastic and fracture characteristics of the bone19,20.

In particular, nearly all of the nanoindentation studies on bio-tissues reported to-date employed the Oliver-Pharr method13 to obtain elastic modulus and hardness values from the nanoindentation data. The basic assumption involved in this method is that the sample behaves purely elastically during unloading, but biological tissues such as bone are well-known to be viscoelastic in both the macroscopic level as well as the microstructural level11. Material viscoelastic effects during unloading are well-known to lead to erroneous results in the estimation of contact stiffness and area using the Oliver-Pharr method13, and in the past, increasing the holding time before unloading and increasing the unloading rate have been suggested as effective procedures to reduce viscoelastic effects during unloading19,20. An alternative solution is to allow the viscoelastic effects to occur. But then a method that has been well established in monolithic engineering materials to correct for the viscoelastic effects should be used21.

This study is focused on the use of nanoindentation as a tool for quantification the differences between micro mechanical properties of femoral cortical bone of healthy laying hen and laying hen with defective calcic metabolism.

2. Materials and methods

2.1. Hens' femoral bones

Two bone tissues were compared, both belonging to Rhode Island Red (RIR) laying hen, caged and fed in identical conditions in the breeding station in the Czech Re-
The birds were kept in the three-floor cage housing, with 650 cm² floor space of individual housing. One of the tissues was extracted from the cortical part of a femoral bone of clinically healthy hen (51 weeks old) with the incidence of cracked eggs lower than 2% (further denoted as Healthy-series). The second one was extracted from the cortical part of a femoral bone of a hen with calcium metabolism defect (denoted as Ill-series). This defect was shown by a high presence of cracked eggs (more than 20%).

2.2. Preparation of specimens

The bone specimens were dissected from the femoral diaphysis of a mature hen (RIR) and dried for 48 hours at room temperature. Effect of the bone drying and affecting the values of Young's modulus and hardness was documented for bovine bones but not for hen's bones so far. The samples were milled down to a cylindrical shape of 10 mm in height, their main (cylindrical) axis being aligned with the longitudinal direction of the diaphysis. The specimens geometry and different stages of the testing procedure are shown in Fig. 1. After this preparatory step, the specimens were cold-prepared (the structure was not thermally affected). 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.

2.3. Experimental set-up and loading conditions

The experiments were performed using nanohardness tester (CSM Instruments, Switzerland). A standard Berkovich tip was brought to the sample surface, producing a series of imprints. Influences of the tip geometry, contact depth, and contact area on nanoindentation properties of the bone were broadly discussed in literature, and the results were used for the configuration of presented experiments. The indenter has a nominal tip radius of $R \approx 50$ nm and a half-angle apex of $\theta = 65.27^\circ$. The bone fragments were loaded in directions perpendicular to the cross-sections. Load vs. depth of penetration was measured throughout the whole procedure of loading, holding, and unloading. The load-controlled test was performed using the standard trapezoidal loading diagram as follows: linear loading $(60 \text{ mN min}^{-1})$ up to the peak force $(5 \text{ mN})$, then a 10 s holding period at the maximum force and linear unloading $(60 \text{ mN min}^{-1})$ to zero force level (Fig. 2). Each sample cross-section was covered with a grid of 80 indents with $12 \mu m$ spacing. Similar experimental procedure and set-up was used e.g. by Severa et al.17.

3. Results

Elastic modulus $(E)$ and indentation hardness $(H)$ were evaluated by standard procedure from unloading branches of a loading diagram for each indent. Although, the elastic parameters show high scatter easily distinguishable decrease in both $E$ and $H$ can be observed for Ill-series as depicted in Figs. 3 and 4.
The results of the performed experiments revealed that bone of a clinically healthy hen exhibited higher elastic modulus \( E \) and indentation hardness values \( H \). It points to the fact that both elasticity and strength parameters (that are related to \( H \)) are affected in III-series. In case of non-defective tissue, following values were determined as:

\[
E = 27.5 \pm 2.8 \text{ GPa}, \quad H = 0.99 \pm 0.11 \text{ GPa},
\]

while in case of defective tissue as:

\[
E = 24.5 \pm 3 \text{ GPa}, \quad H = 0.88 \pm 0.07 \text{ GPa}.
\]

Generally speaking, the values measured on the defective bone are approximately 11% lower.

Based on optical microscopical observations, the healthy bone tissue contained larger pores in comparison with defective one. Interpretation of this fact needs much deeper investigation.

### 4. Discussion

This study utilised nanoindentation to investigate the mechanical properties of the microstructure of bone from the mid-femoral diaphyses of healthy and defective laying hen. Unlike the more conventional microhardness techniques, nanoindentation provides both modulus of elasticity and hardness estimates for a material and can be used to target specifically various bone tissue structures at a microscopic level.

A strong motivation behind the use of nanoindentation is the potential to understand the mechanical competence of a whole bone in the light of the properties of its structural units (osteons and trabecular packets) that result from the ongoing remodeling activity. However, given the prohibitively wide range of elastic data measured using bending, buckling, tensile, ultrasound tests or calculated using inverse numerical techniques\(^{27}\), it still remains unclear how the indentation modulus can be converted into the elastic response of the bone tissue measured at higher levels of its hierarchical organization\(^{28,29}\).

In general, the variations that have been recorded in the elastic modulus at the bone matrix level are rather high. The elastic modulus ranged between 22 and 38 GPa in osteonal bone of healthy hen and 16 and 32 GPa in osteonal bone of defective hen. The large scatter in \( E \) (as well as \( H \)) can be attributed mainly to the naturally varying crystallographic orientations within the cross section of the bone (differently oriented anisotropic lamellas and its fibers)\(^{30,31}\). The shape of the \( E \) and \( H \) histograms shows also on some kind of bimodality in the case of Healthy-series which could be related to the prefferential collagen fiber orientations in the tested area. Despite this fact, presented study was focused on the evaluation of average properties in one direction only to give overall insight into the problem. More detailed research is planned in the future.

### 5. Conclusions

In the current study the differences between healthy and defect cortical bone tissues were documented and analysed in an average sense. Elastic modulus and hardness dropped down by ~11% for defected series. Similar approaches as used for studying human bones were employed. The data revealed and confirmed the fact that mechanisms described for human tissues can be largely adopted and used for detailed further research of hens' bones as well.

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L. Severa*, J. Němeček*, L. Mácha†, J. Votava*, and J. Buchar* (MENDELU, Brno, † CTU in Prague, Czech Republic): Nanoindentation Based Microanalysis of Hens’ Bones

The research is focused on the use of nanoindentation as a tool for quantification of the differences between micro mechanical properties of femoral cortical bone of healthy laying hen and laying hen with defective calcic metabolism. In general, the variations that were recorded in the elastic modulus at the bone matrix level are rather high. The elastic modulus ranged between 22 and 38 GPa in osteonal bone of healthy hen and 16 and 32 GPa in osteonal bone of defective hen. The shape of the $E$ and $H$ histograms shows also some kind of bimodality, which can be related to prefferential collagen fiber orientations in the tested area.
Galvanic Alloys for Protection Against Corrosion

1. Introduction

Galvanic alloys are used for protection against corrosion because they are more resistant to corrosion than the base metal.

2. Experimental procedure

The following procedures were used for the preparation of the samples:

- Prealloyed Astaloy85Mo powder (Höganäs AB),
- Atomized ASC100.29 powder (Höganäs AB),
- Manganese in form of medium carbon ferromanganese (80 % Mn, 1.1 % C, particle size < 45 μm, 0.67 % O, milled in air, EratemElkem),
- Natural graphite CR12 (Grafit Netolice).

The powders were mixed as two systems:

A) Fe – 0.85 % Mo – 3 % Mn – 0.5 % C (referred to as A)
B) Fe – 3 % Mn – 0.5 % C (referred to as B), both were prepared with 0.8 % HW wax as lubricant. The samples (Ø 10 × 10 mm) made of the mixed powders compacted at 600 MPa were sintered in dissociated ammonia (dew point –30 °C) at 875 °C, 1120 °C and at 1200 °C for 1, 3, 5, 10, 30 and 60 min in a Mars furnace.

3. Results and discussion

The aim was the comparison of microstructure creation for iron and molybdenum powders independence on manganese addition at chosen sintering conditions, i.e. ferrous matrix alloyed by molybdenum and matrix without molybdenum alloying. Microstructure of samples for both systems A and B was covered after sintering for 1 min with a thin manganese alloying layer as a result of sublimation and condensation of manganese vapors.

After sintering at 1200 °C for 3 min, Fig. 1a, the manganese was alloyed by manganese along grain boundaries – intensive bounders (diffusion to be at grain boundary faster than in a volume). It came to further highlight of grain boundaries and partly to alloying of core particles after sintering within 5 min. Character of microstructure samples of system B after sintering 30 min corresponds approximately to microstructure samples according to Fig. 1b. In system A, sintered at 1120 °C during 1 min, we observe more expressive alloying only on particles surface. On the other side system B alloying of core particles by manganese is more intensive at grain boundaries. This shows that the diffusion of manganese from surface was quicker due to higher sintering temperature in molybdenum alloyed matrix. Microstructure of this system was heterogeneous, but evidently different. Alloying of matrix extends in both systems after sintering for 3 min at sintering 1120 °C, but more in molybdenum alloyed.

After sintering at 1200 °C perhaps equal behavior of alloying matrix is observed but only with the difference that a higher homogeneity value of microstructure was achieved. Microstructure of system A evidently consists of bainite and manganese supports bainite formation especially after sintering at 1200 °C for 60 min.

Microhardness values for both systems are described in Fig. 2. Higher microhardness values were in samples...
prealloyed by molybdenum, Fig. 2a. Microhardness of edges particles at samples of B system after sintering at 875 °C was possible measured during sintering for 30 min only. This also shows the slower diffusion of manganese in iron matrix. It is clear that microhardness values were also affected by carbon in border areas. The microhardness values of microstructure samples sintered at 1120 °C are shown in Fig. 2b. This diagram evidently shows continuous increase of microhardness at borders and small change of microhardness in centre of elements systems. On basis of high microhardness value it can be expected that in border of manganese alloyed areas carbides type of (Fe-, Mo-, Mn-) C were created. The microhardness values of both types of samples after sintering at 1200 °C are shown in Fig. 2c. These microhardness values again show the molybdenum prealloyed matrix as uniform and more hardened, which can be caused as we already mentioned by carbides formation. Microhardness of grain borders corresponds to a martensite after longer sintering time.

4. Conclusions

Following main results were obtained: Mo alloyed and Fe matrix of compacts samples are alloyed by manganese already at low temperature and apparently during a 3 min. By extension of sintering time and by higher sintering temperature the alloying process of matrix by manganese is reflected by the alloying of powder elements inside and by bigger or smaller homogeneity. We have found out in molybdenum alloyed matrix a more uniform alloying by manganese, which was demonstrated by higher microhardness values. Ferritic grains were located in iron matrix after longest sintering time and at higher sintering temperature. Microstructure of investigation material on bases of prealloyed powder affected by manganese comprised mainly bainite and the grains of ferrite occurred in microstructure of mixed system under all sintering condition.

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V. Simkulet, and L. Parilák (Faculty of Manufacturing Technologies of the Technical University of Košice with a seat in Prešov, Slovakia): Microstructures Characteristics of Fe-0.85Mo-3Mn-0.5C Sintered Steel in Dependence on Sintering Conditions

Manganese in combination with Molybdenum atomized prealloyed powder forms a new group of sintered high strength steels. The final properties of these steels depend on microstructure homogeneity. The aim was to investigate the alloying of molybdenum prealloyed powder with manganese in comparison with plain iron powder. The circular cross section samples were prepared for the investigation from basis water atomized and plain iron powders. The microstructure characteristics of sintered samples were characterized by microhardness measurement.
COMPARISON OF MECHANICAL PROPERTIES OF NITRIDED CASES AND REMELTED LAYERS OF AUSTENITIC STAINLESS STEEL

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Keywords: remelted layer, nitrided layer, austenitic stainless steel, diffusion layer, termochemical treatment, glow-discharge nitriding process, nanohardness

1. Introduction

The austenitic stainless steels have very high general corrosion resistance, but they have low hardness and wear resistance. Nowadays, material technologies like laser remelting and low-temperature glow-discharge nitriding process can improve mechanical properties of austenitic stainless steel without decrease the corrosion resistance. Laser remelting influence on refinement of microstructure and homogenizing of chemical composition of alloys was studied in ref1–4. If the austenitic stainless steels are subjected to glow-discharge nitriding process in low temperature, loss of corrosion resistance is not observed. At the temperatures above 450 °C, the precipitation of CrN was observed. The limiting temperature of nitriding process could be about 450 °C, according to the5–8.

The aim of this article is an analyse of local mechanical properties of nitrided cases and remelted layers after the low temperature glow-discharge nitriding process and laser remelting, respectively.

2. Experimental procedures and results

Every test specimens (diameter of 20 mm and height of 6 mm) were made of austenitic stainless steel type X5CrNi18-10. Young’s modulus of the steel substrate is approximately 200±14 GPa and its hardness is about 220±3 HV20. The specimens were subjected to glow-discharge nitriding process at the temperature of 450 °C. Chemical composition of gas mixture during the termochemical treatment was different. Parameters of nitriding process are shown in Tab. I.

The same steel type X5CrNi18-10 was subjected to laser remelting. Laser remelting was done by means of laser MLT 2500 CO₂ (wavelength 10.6 μm) in argon atmosphere. During the laser remelting process no. 5 and 6 the specimens were also immersed in liquid nitrogen. The laser beam dimension 1x20 mm was used. Tab. II presents parameters of laser treatment.

Representative picture of remelted layers is presented in Fig. 1. After laser remelting the surfaces were grinded off through 1200 grit SiC papers. The depth of the remelted layers depended on the laser power. Generally, the increase of the power caused a rise of thickness of the remelted layers.

Investigation of mechanical properties was carried out by using hardness tester with mounted Berkovich indenter. Mechanical properties of the diffusion layers were

<table>
<thead>
<tr>
<th>Number of process</th>
<th>Scanning velocity [m min⁻¹]</th>
<th>Power [kW]</th>
<th>Scanning velocity [m min⁻¹]</th>
</tr>
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<tbody>
<tr>
<td>3</td>
<td>2</td>
<td>2</td>
<td>0,25</td>
</tr>
<tr>
<td>4</td>
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<tr>
<td>6</td>
<td>2</td>
<td>5</td>
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Table I

Parametres of the glow-discharge nitriding process

<table>
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<tr>
<th>Number of process</th>
<th>Vacuum pressure [hPa]</th>
<th>Time [h]</th>
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<th>Atmospheric composition</th>
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<td>6</td>
<td>450</td>
<td>50</td>
<td>50</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 1. Microstructure of remelted layer after process laser remelting
examined on the surface of specimens using a different loads. Results of surface hardness were obtained by using different loads, respectively: 10, 20, 30 mN. Research was done by means of CSEM NanoHardnessTester (NHT) produced in Switzerland. Oliver-Pharr method was used for calculation of modulus. The value of Poisson’s ratio was 0.3. Nanohardness was automatically recalculated between scales and presented in Vickers hardness scale. The results are shown in Tab. III.

### 3. Conclusion

1. Glow-discharge nitriding process has beneficial influence on nanohardness and Young’s modulus.
2. The increase of nitrogen content in gas mixture influence on higher value of Young’s modulus.
3. Laser remelting caused the refinement of microstructure in obtained surface layer.
4. Laser remelting process has beneficial influence on nanohardness and Young’s modulus.

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A. Sitoiko, M. Szkodo, and B. Sniegocka (University of Technology in Gdansk, Faculty of Mechanical Engineering, Department of Materials and Welding Engineering, Poland): **Comparison of Mechanical Properties of Nitrided Cases and Remelted Layers of Austenitic Stainless Steel**

This article presents the results of nanohardness and Young’s modulus of nitrided cases and remelted layers. The nitried cases were obtained by using the glow-discharge nitriding process at the temperature of 450 °C. The thermochemical treatment was done by using a different chemical composition of gas mixture (N2:H2). The laser remelting was done by using the TRUMPF laser TLF 6000 turbo in Kielce. The laser remelting was done by using different parameters of thermochemical treatment. Investigation of mechanical properties was carried out by using hardness tester with mounted Berkovich indenter. Mechanical properties of the diffusion and remelted layers were examined on the surface of specimens using different loads.
NANOINDENTATION TESTING OF LOW-ALLOYED MOLYBDENUM SINGLE CRYSTALS

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Keywords: nanoindentation, molybdenum alloy, single crystal, hardness, elastic modulus

1. Introduction

Nanoindentation methods are applied to study nano-scale material deformations and enable accurate measurements of indentation load \( P \) and penetration depth \( h \). Mechanical properties, Young’s modulus and hardness can be obtained from nanoindentation by numerical calculations of the load-displacement curves including loading and unloading process according to Oliver-Pharr method (ref.1–5). Recent research has shown that the hardness determined by nanoindentation depends on a test load, i.e. hardness at small depth is much greater than at greater depth. This phenomenon is called the Indentation Size Effect (ISE) (ref.5,6). Another method used to measure hardness from load-depth curves is work-of-indentation method. The total mechanical work of indentation can be determined by computing the area under the force increasing and force decreasing curve.

For sharp indentation of elastic-plastic material, the loading response is governed by \( P = Ch^2 \). The plastic work \( W_{pl} \) can be determined from Eq. (3):

\[
\frac{W_{pl}}{W_{t}} = 1 - \frac{h_f}{h_{max}}
\]

where \( h_f \) is the final depth of contact impression after unloading and \( h_{max} \) is the indenter depth at peak load. The hardness concerning total work of indentation, resp. plastic work of indentation can be calculated from Eq. (4) (ref.5), where \( \kappa \) is a constant equal to 0.0408 for the three sided Berkovich pyramidal indenter.

\[
H_{wt} = \frac{\kappa P_{max}^3}{9W_t}, \quad \text{resp.} \quad H_{wpl} = \frac{\kappa P_{max}^3}{9W_{pl}}
\]

Atomic force microscopy (AFM) can be used for imaging of residual indentations and obtaining accurate dimensional information from an image area of only a few microns.

In this paper, the indentation hardness, Young’s modulus, total and plastic work of pure and low-alloyed molybdenum single crystals are investigated. We compare hardness from the Oliver and Pharr analysis (\( H_{OP} \)) and the work-of-indentation method described above. The influence of niobium as an alloying element and crystallographic orientation of single crystals on these nano-scale properties is studied. The effect of the load on pile-up formation is also investigated using AFM imaging.

2. Experimental

Bulk single crystals of Mo-2 wt.% Nb with crystallographic orientation \(<110>\) and \(<100>\) were used for the experiment. The pure molybdenum single crystal with crystallographic orientation \(<110>\) was included in the experiment due to a study of the influence of niobium on the mechanical properties of molybdenum single crystal. All single crystals were prepared by the electron beam zone melting (method of floating zone). The surface of specimens was polished using diamond pastes and electrolytically in NaOH solution. The indentation experiments were conducted with the standard three-sided pyramidal Berkovich tip using Triboindenter TI 950 (Hysitron). The tip radius was about 150 nm. For an easier interpretation of mechanical behaviour at various depth, the maximum load...
was changed at intervals: 1000, 2000, 3000 and 4000 µN and then unloaded. The dwell time was 2 s. For each material, six indentations were made at each load and the presented results are averages for the group. These indentation loads correspond to applied depths from 69 to 190 nm for all specimens. The topography of the selected indents was acquired using atomic force microscope SOLVER NextTM (NT-MDT) operated in contact mode. The probes PPP-CONTR (Nanosensors) were used for imaging. The AFM images were processed using software Gwyddion (version 2.25).

3. Results and discussion

The load-depth curves for Mo-2 wt.% Nb <100> single crystal for all applied loads are given in Fig. 1 as an example.

The elastic recovery appears in load-depth curves during unloading processing for all specimens, but the elastic recovery of Mo-2 wt.% Nb with crystallographic orientation <100> is less than other specimens. Fig. 2 shows one of AFM 3D-images of indentations (load 4000 µN) for molybdenum-base single crystals. It is visible that edges of an indentation bend to center because of elastic recovery.

The hardness values $H_{IT}$, $H_{WP}$ and $H_{WP}$ of all specimens are plotted as a function of applied load in Fig. 3 to 5. The results display a strong size effect, i.e. the hardness decreases as the indentation load or indentation depth increases, which is commonly referred to as the indentation size effect (ISE). Numerous investigators have reported studies of ISE using nanoindentation testing for various materials (ref. 6,10,11). The hardness values calculated using the plastic work of indentation are the highest, especially in smaller depth or for low applied loads. It was found that the hardness of all specimens estimated by the O-P method was significantly (up to 30%) lower than the hardness calculated using the total work-of-indentation. There are only small differences between the values of hardness for both specimens of Mo-Nb single crystals with different crystallographic orientation. The plastic work must be less than total work, but the plastic hardness $H_{WP}$ is larger than $H_{WP}$.

The hardness values calculated from the work-of-indentation approach rise more steeply at lower applied load than the hardness values calculated by the O-P method. The reason of this effect can be explained by the fact that the method of Tuck et al. (ref. 9) makes no allowance for changes in tip geometry at lower indentation depths, where the tip geometry can significantly influence the calculated values, whereas tip geometry effects are allowed for in the O-P calculations.

At low loading, the indent behaviour of specimens is almost elastic deformation, increasing the Young’s modulus ($E_{IT}$) – Fig. 6. It was observed that the hardness and Young’s modulus of Mo-2 wt.% Nb single crystal with the
orientation <100> increased at the indentation load 4000 µN probably results of the hardening effect (ref.6).

The contribution of the elastic work to total work decreases with increasing maximal load approx. from 21 to 17 % in the case of Mo-2 wt.% Nb and pure Mo single crystals with the orientation <110>. Because of the smaller elastic recovery for indentations into Mo-2 wt.% Nb <100>, there is lower differences between the total work of indentation approach and the plastic work of indentation approach – see Fig. 7.

There is the evident influence of crystallographic orientation of single crystals on mechanical properties. It is known that single crystals of molybdenum and its alloys show the anisotropy of mechanical properties, when the crystals with the crystallographic orientation <110> have much higher plasticity than these crystals with the crystallographic orientation <100> (ref.12). The Mo-Nb single crystal with orientation <100> has higher values of Young’s modulus and $H_{\text{IT}}$ at most testing loads than adequate alloy with orientation <110>. The similar effect was observed in case of macro-scale testing of mechanical properties of Mo-Nb single crystals (ref.12). The alloying of molybdenum single crystal with niobium results in the increase of Young’s modulus and hardness.

The pile-up formation was visible in the AFM images of the indents only for Mo-2 wt.% Nb single crystal with orientation <100> (Fig. 8).

This result corresponds to findings by Stelmashenko et. al. (ref.13). In their study, the height of pile-ups on the (110) surface in molybdenum single crystal was very small for each of three different orientations of indenter, and there is no symmetry in the pile-up distribution.

4. Conclusions

The experiments and the above discussion yield the following results:

a) Under the same test condition, the nanohardness and Young’s modulus of all specimen declines as the load increases. The ISE of hardness values was observed.

b) The hardness estimated by the O-P method was significantly lower than the hardness calculated using the total work-of-indentation approach. The main advantage of work-of-indentation method is that there is no need to calculate the area of the indentation, which
thus eliminates the problems caused by underestimating the contact area.

c) According to literature findings (ref.12,13), the hardness values should be higher for molybdenum-base single crystals with crystallographic orientation <100> than for these with crystallographic orientation <110>. The calculated HWt and HWpl values for the tested Mo-Nb single crystals don’t correspond to this fact. Thus the discrepancy of these hardness values must be examined.

d) Pile-up formation occurred at the edges of the inden- tations only in Mo-2 wt.% Nb single crystals with crystallographic orientation <100>. The widely used Oliver-Pharr model does not account for pile-up and consequently can overestimate hardness and elastic modulus.

e) It was confirmed that mechanical properties depend on the crystallographic orientations of single crystals. According to the results, the Mo-Nb single crystal with orientation <100> has higher values of EIT than the same single crystal with orientation <110>. The alloying of molybdenum with niobium led to increasing of values of hardness and EIT.

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K. Skotnicová a, M. Vyležík b, V. Matějka c, and J. Drápal a (a Department of Non-ferrous Metals, Refining and Recycling, VSB – Technical University of Ostrava; b Centre for Advanced Innovation Technology, VSB – Technical University of Ostrava; c CNT – Nanotechnology Centre, VSB – Technical University of Ostrava, Czech Republic): Nanoindentation Testing of Low-Alloyed Molybdenum Single Crystals

The indentation hardness, Young’s modulus, total and plastic work of pure and low-alloyed molybdenum single crystals were investigated. It was found that the hardness of specimens decreases as the indentation load or indentation depth increases due to the indentation size effect (ISE). The same trend was observed for the EIT values of all specimens. The hardness estimated by the O-P method was significantly (up to 30 %) lower than the hardness determined using the total work-of-indentation approach. AFM 3D-images of indentations showed that pile-up formation occurred at the edges of the indentations only in Mo-2 wt.% Nb single crystals with crystallographic orientation <100>. It was confirmed that nanoscale mechanical properties depend on the crystallographic orientations of single crystals. The alloying of molybdenum with niobium led to increasing of values of hardness and EIT.
INHOMOGENEOUS PLASTIC DEFORMATION OF TINPLATES UNDER UNIAXIAL STRESS STATE

EMIL SPIŠÁK, JÁN SLOTA, JANÁ MAJERNÍKOVÁ, L’UBOŠ KAŠČÁK, and PETER MALEGA

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Keywords: tinplate, plastic deformation, failure analysis

1. Introduction

Tinplate is essentially low carbon steel, cold reduced between 0.13 and 0.50 mm thick coated with tin produced for packaging industry. Modern tinplate possesses several important advantages, such as excellent drawability combined with good strength, good solderability and corrosion resistance. In many instances, special grades for specific can making techniques have been developed, e.g. for drawing and wall-ironing, redrawing and easy-open ends. Their development has required close cooperation between packaging producers and tinplate manufacturers.

During the cold rolling on tandem mill the strength and hardness have been increased. On the other hand, the plastic properties of rolled materials have been decreased. To eliminate these negative changes in material, the recrystallization annealing is included in the sheet production process. Some products may be either batch annealed (BA) or continuously annealed (CA). Although the temper of the plate will be the same the mechanical properties may differ as CA plate has a finer grain structure.

A number of materials exhibit discontinuous yielding under monotonic tension. A typical example is annealed mild steel. The monotonic stress–strain curve is not smooth but shows marked irregularities, with negative slopes occurring at or near the initial yield. Elastic deformation is terminated at a stress level known as the upper yield stress. Deformation proceeds at a decreased stress level known as the lower yield stress accompanied with inhomogeneous deformation. The specimen is divided into regions where the strain is relatively high (Lüders strain) and regions which are still elastic. The distinct plateau in the stress–strain curve is characterized by the propagation of Lüders bands, the elastic zones are essentially free of dislocations whereas Lüders bands have a high dislocation density. The upper yield stress is regarded as the nucleation stress, and the lower yield stress is the growth stress of the Lüders bands.

The propagation of Lüders bands is influenced by many factors including crystal structure, grain size, composition and microstructure, shape and stiffness of the testing sample, strain rate, and the type of loading. Grain size has a great influence on the Lüders strain and the morphology of the Lüders bands, particularly in the case for mild steels. The Lüders strain decreases significantly as the grain size increases. Zhang and Jiang experimentally studied the local plastic deformation of a carbon steel subjected to monotonic tension. It was found that the strain at the Lüders front was lower than the full Lüders strain (the length of the plateau on the stress–strain curve). During the propagation of Lüders bands, the local deformation is inhomogeneous. The local strain was inhomogeneous even at the work-hardening stage.

In the paper, the BA and the CA double reduced tinplates for can making industry have been analysed. Inhomogeneous plastic deformation of DR tinplates under tension loading was experimentally studied.

2. Experiments

Tinplates are currently produced mainly two ways of rolling. Thicker steel sheets (0.18–0.30 mm) are produced by single reduction, after which the plates are continuous annealed. The sheets of smaller thickness (0.135–0.18 mm) are after the single reduction and annealing a second time rolled (double reduced – DR). Most of the current packaging sheets are further processed by
drawing (drawing two-pieces containers, lids, twist caps, etc.). For this reason, the thin sheets of packaging have to meet certain requirements on the mechanical and plastic properties. Compliance with mechanical properties that are mainly characterized by the yield stress and the tensile strength, it is currently difficult to achieve by the manufacturer within the required limits. Significantly greater problem is the plastic properties of thin metal packaging and method of their evaluation. At present the evaluation of thin sheets for packaging (in terms of standards) mainly the tensile test is used, but on the basis of supplier – customer relations are often used other tests (Springback test, Erichsen cupping test, Bulge test, and others). Based on past experience, the tensile test seems to be problematic for evaluation of thin steel packaging DR sheets9,10.

In this work, a double reduced tinplates of TH550CA and TS550BA, respectively, with thickness of 0.17 mm were used for experiments. To determine an anisotropic properties of tested materials for the uniaxial tensile test samples in rolling direction 0° and perpendicular direction 90° in respect of rolling direction have been taken. From the uniaxial tensile test the following parameters have been evaluated: the yield stress, the ultimate tensile strength and total elongation.

The measured values are shown in Table 1. Typical chemical compositions for the tested materials are given in Table II.

Microstructures of the investigated steels in the direction of 0° and 90° are shown in Fig. 1 and Fig. 2. For both annealing processes of tested thin steel sheets the failure zones are shown in Fig. 3 and 4.

For these sheets that show Lüders band slip at uniaxial tensile test it is problematic to determine the value of maximum uniform deformation10. On tested samples it is showed a strain creation in specific sample sections. It starts in one place, suddenly stops and passes into a completely different sample place (see Fig. 4). During the propagation of Lüders bands, multiple Lüders fronts can be formed. Under tension with a constant axial load, the Lüders front was approximately parallel to the material plane of maximum shear stress4.

As for batch annealed sheets there have been ruptures in all samples during local sheet strain without any expansion of strain in the whole measured length of tested samples (see Fig. 3 and 4).

In Figs. 5 and 6, the surface failures of tested samples are shown. From Fig. 5a) we can expressly conclude that except primary slip planes where the rupture of tested samples appeared, also the so called secondary slip planes appeared in their proximity.

Local thinning of tested sheet has occurred in these places, as well. In the rest of measured part the tested sample has not been plastically deformed. The rupture surface in Fig. 5a, but also the detail in Fig. 5b and in Fig. 6 show, that in the place of sample rupture a sharp contraction (necking) has occurred. It points at the fact that the materi-

<table>
<thead>
<tr>
<th>Sample</th>
<th>Uniaxial test</th>
<th>Biaxial test</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Rp0,2 [MPa]</td>
<td>Rm [MPa]</td>
</tr>
<tr>
<td>TS550BA ⊥</td>
<td>442</td>
<td>434</td>
</tr>
<tr>
<td>TS550BA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TH550CA ⊥</td>
<td>538</td>
<td>563</td>
</tr>
<tr>
<td>TH550CA</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table II
Chemical composition of experimental materials

<table>
<thead>
<tr>
<th>Composition</th>
<th>C [%]</th>
<th>Mn [%]</th>
<th>P [%]</th>
<th>S [%]</th>
<th>Si [%]</th>
<th>Cu [%]</th>
<th>Al [%]</th>
<th>Cr [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS550BA</td>
<td>0.081</td>
<td>0.41</td>
<td>0.018</td>
<td>0.003</td>
<td>0.006</td>
<td>0.041</td>
<td>0.04</td>
<td>0.02</td>
</tr>
<tr>
<td>TH550CA</td>
<td>0.055</td>
<td>0.17</td>
<td>0.018</td>
<td>0.002</td>
<td>0.008</td>
<td>0.036</td>
<td>0.05</td>
<td>0.02</td>
</tr>
</tbody>
</table>
al itself has better plastic properties than the plastic properties measured by method of elongation at uniaxial tensile test.

3. Experimental results and discussion

Mean value of elongation of the sheets from 1 to 3 % was measured. Significant differences in mechanical properties were observed in the direction of 0° and 90°. In most cases, the difference of elongation was about 100 %. The results of mechanical tests showed that the uniaxial test of tinplates, especially double reduced, does not provide a true representation of their plastic properties. This fact is fully reflected in cupping test when the cups from packaging steel sheets with an elongation from 1 to 3 % were produced (LDR = 1.67) (ref.7–9). Despite very low values of the elongation, it was found that the failure is typical plastic (Fig. 7) and there is a large contraction (Fig. 5). From the details of fracture surface in Fig. 5b, but mainly from Fig. 6, we can clearly see the slip planes which are observed on the surface of the sheet near the fracture as waves. Thus, these tinplates have the local plasticity, but either the plastic strain is inhomogeneous on the whole gage section of the sample or plastic strain propagates only in local band and continue to failure. This cross-section is not able to transfer the strength necessary for the strain of another section of tested sample. The fracture surface shows characteristic signs of plastic intercrystalline fracture expanding along grain boundaries where inclusions can be found.

A general concept is that the end of the plateau is the starting point of homogeneous deformation in the gage section. However, the results shown that at the end of the plateau, the local deformation was still inhomogeneous. The inhomogeneous deformation persisted in the work-hardening stage. This observation is consistent with that observed by other researchers4,6. It was found that the local axial strain increased linearly and rapidly with time, indicating the propagation of the Lüders front over the area. A large amount of ferrite grains within this area experienced plastic deformation. For a mild steel, authors7 pointed out that the strain rate at the Lüders front was rather high and some dislocations unable to move at the high strain rate can continue to move at a slow velocity after the front has passed them. Some deformed ferrite grains may deform further and some undeformed ferrite grains may experience a delayed plastic deformation. Such actions result in creep deformation. Similar phenomenon was observed in a monotonic tension of a mild steel8 and an aluminum alloy8.

Localization of deformation and fracture in a tensile test samples can be explained by the Marciniak theory,
whereby the localization of deformation occurs in areas with material inhomogeneity. Inhomogeneity can be represented by changing the microgeometry of the surface of the material or inhomogeneity (inclusions, cracks caused by one particular reduction of the grain boundaries). Failure of samples is probably initiated by the creation of the local neck in a certain place of the sample. This results in earlier failure and lower ductility. Thus, so narrow region cannot further transmit escalating loading and failure occurs just in this area.

4. Conclusions

In this paper, the causes and diversity of inhomogeneous strain were studied. The failure of tinplates during plastic deformation of simple uniaxial test sample was analysed. Experiments showed that plastic deformation of the samples during loading occurs only in certain places. This means that the plastic deformation do not extend to whole volume of the sample. On the other hand, the analysis of the fracture surface indicates that there is no brittle fracture. From achieved contraction of the sample it is clear that ductile fracture is concerned. This phenomenon can be attributed to inhomogeneity of the material structure, while the way of annealing of the material and structural inclusions, respectively, influence behaviour of the tinplates during loading. One of the conclusions is that there is usually either a one slip plane (TH550CA) with localization of the deformation in the slip plane up to failure or more slip planes (TS550BA) with Lüders bands. More slip planes may be related to the fact that microstructure of the TS550BA tinplate include a larger grains.

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E. Spišák, J. Slota, J. Majerníková, I. Kaščák, and P. Malega (Faculty of Mechanical Engineering, Technical University of Košice, Košice, Slovak Republic): Inhomogeneous Plastic Deformation of Tinplates under Uniaxial Stress State

Tinplates are mainly processed by forming nowadays. It is necessary to know their properties for the evaluation of their suitability for the forming processes. The paper deals with the inhomogeneous plastic deformation during uniaxial loading and the localization of plastic deformation which lead to the early failures of the tinplates. Causes of inhomogeneous strain and local propagation of deformation were analyzed.
JOINING MATERIALS USED IN CAR BODY PRODUCTION BY CLINCHING

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Keywords: clinching, car body sheets, evaluation of properties

1. Introduction

The automotive industry is currently working to accommodate the conflicting requirements of both environmental legislation and customer demands for greater performance and more luxury and safety features, by developing a light-weight and therefore essentially, energy-efficient vehicle. One of the possibilities of decreasing the car weight and consequently lowering the fuel consumption is using various combinations of materials, such as combination of conventional deep-drawn steel sheet and high-strength steel sheet. In the areas, where high passive safety is needed, high-strength steels such as TRIP can be used. The usage of such steels can significantly reduce the car weight.

The increasing use of coated, lightweight and high-strength materials has led the automotive industry to re-examine traditional methods of component assembly. For example, direct welding of dissimilar sheet metals has proven to be difficult or impossible; thus, alternative joining techniques, such as mechanical fastening systems, have attracted increasing interest and applications in recent years. Mechanical fastening encompassess a broad range of methods, from threaded fasteners to different forms of rivets and mechanical interlocking methods.

One of these methods is clinching technology, which has not attracted much attention from researchers as yet, so it has not been studied deliberately so far. Clinching does not use any kind of appending joining components (such as screws, bolts).

Only a die and a punch are used to press the sheet components to finish the whole joining process. The clinching process is a combination of drawing and forming that locks together sheets metal layers. The blanks are plastically deformed and the shape of the tools remains theoretically unchanged during the clinching processes. The punch is movable, whereas the fixture and the die are fixed during the process. The punch force needed for the joining process depends on the thickness and the strength of the materials to be joined, the size of the tools and friction coefficient usually varies from 10 to 100 kN (ref.5,6).

The paper evaluates joints made by clinching the following materials: microalloyed steel HSLA H220PD, TRIP steel 40/70+Z100MBO and drawing grade steel DX51D+Z.

2. Materials and methods

The following steel sheets were used for experiments: microalloyed steel HSLA H220PD with the thickness of

<table>
<thead>
<tr>
<th>Material</th>
<th>Rp0.2 [MPa]</th>
<th>Rm [MPa]</th>
<th>A80 [%]</th>
<th>n50</th>
</tr>
</thead>
<tbody>
<tr>
<td>H220PD</td>
<td>238</td>
<td>382</td>
<td>36</td>
<td>0.228</td>
</tr>
<tr>
<td>TRIP 40/70</td>
<td>450</td>
<td>766</td>
<td>26</td>
<td>0.278</td>
</tr>
<tr>
<td>DX51D+Z</td>
<td>≥ 140</td>
<td>270-500</td>
<td>≥ 22</td>
<td>*</td>
</tr>
</tbody>
</table>

*not specified

Table I
Basic mechanical properties of used materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Chemical composition in [%] wt.</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>Mn</td>
</tr>
<tr>
<td>-----------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>H220PD</td>
<td>0.06</td>
</tr>
<tr>
<td>TRIP 40/70</td>
<td>0.204</td>
</tr>
<tr>
<td>DX51D</td>
<td>0.64</td>
</tr>
</tbody>
</table>

V Nb Mo Zr
H220PD 0.002 0.026 0.005 0.001
TRIP 40/70 0.004 0.004 0.008 0.007

Table II
Chemical composition (wt.%) of used materials
0.8 mm, TRIP 40/70+Z100MBO with the thickness of 0.77 mm and DX51D+Z with the thickness of 0.9 mm.

Their basic mechanical properties and chemical composition are shown in Tables I and II. Mechanical properties of DX51D steel were specified by producer.

According to the orientation of punch and die to the position of upper and lower joined material, following combinations of steel sheets for press joining were used:

- **Samples A**: H220PD (δ₀ = 0.80 mm) and TRIP (δ₀ = 0.77 mm)*
- **Samples B**: TRIP (δ₀ = 0.77 mm) and H220PD (δ₀ = 0.80 mm)*
- **Samples C**: H220PD (δ₀ = 0.80 mm) and H220PD (δ₀ = 0.80 mm)
- **Samples D**: TRIP (δ₀ = 0.77 mm) and DX51D (δ₀ = 0.90 mm)*
- **Samples E**: DX51D (δ₀ = 0.90 mm) and TRIP (δ₀ = 0.77 mm)*

(*sheet on the die side of press joining tool)

The samples with dimensions of 40 × 90 mm and 30 mm lapping according to STN 05 1122 standard were used for the experiments (Fig. 1). Six samples were prepared for every combination of sheets. It is not necessary to clean the surfaces of samples before clinching.

Clinching was performed on the tension machine ZD 40 made by Werkstoffprüfmaschinen Leipzig Company with the loading range of 40 kN. The force needed for joining was 30 kN. The carrying capacities of the clinched joints were evaluated according to standard STN 05 1122 – Tensile test of spot welded joints. This test was used for measuring the maximum carrying capacities Fmax of the clinched joints. The test was carried out on the metal strength testing machine TIRAtest 2300 produced by VEB TIW Rauenstein, with the loading speed of 8 mm min⁻¹.

Further tests for quality evaluation of clinched joints included the metallurgical analysis and microhardness analysis according to STN EN ISO 6507-1 standard. Microhardness analysis was performed on the sample C with HSLA H220PD sheets.

The results of carrying capacities of clinched joints were compared with the carrying capacities of resistance spot welded joints.

### 3. Results

The measured values of carrying capacities of clinched joints after tensile test in comparison with the measured values of carrying capacities of resistance spot welded joints are shown in Table III. The resistance spot welded joints were made with the optimized values of welding parameters. The resistance spot welds of all observed samples reached higher values of carrying capacities in comparison with clinched joints. On average, the clinched joints reached 13% (samples A), 18% (samples C) and 21% (samples E) of carrying capacities of resistance spot welds.

The carrying capacities of samples B and samples D were not measured, because the joints were not successfully made. The upper sheets of both samples (TRIP 40/70 steel) were cut off in the place of the joint and then pressed to the lower sheet (Fig. 2).

The average value of carrying capacities of samples A was 1008 N. The cracks in the TRIP steel were observed on the die side (Fig. 3), which could possibly have a negative effect, especially during dynamic load. The cracks can even decrease the joints’ corrosion resistance. The values

### Table III

<table>
<thead>
<tr>
<th>Number of sample</th>
<th>Carrying capacity Fmax [N]</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Samples A</td>
</tr>
<tr>
<td></td>
<td>CJ</td>
</tr>
<tr>
<td>1</td>
<td>939</td>
</tr>
<tr>
<td>2</td>
<td>985</td>
</tr>
<tr>
<td>3</td>
<td>1016</td>
</tr>
<tr>
<td>4</td>
<td>1080</td>
</tr>
<tr>
<td>5</td>
<td>1093</td>
</tr>
<tr>
<td>6</td>
<td>937</td>
</tr>
</tbody>
</table>

CJ – clinched joints, RSW – resistance spot welded joints
of carrying capacity of samples A are similar to the values measured in clinched joints of the common drawing grade steel sheets, as was published in\textsuperscript{6}.

The average value of carrying capacity of samples C was 970 N. No cracks occurred in the place of the joint from the side of the die. The carrying capacity values of samples C are similar to the values measured in clinched joints of common drawing grade steel sheets.

The average value of carrying capacity of samples E was 1578 N. Cracks in the TRIP steel on the die side were observed, similar to those in sample A (Fig. 3). The measured values of carrying capacity of samples E are higher than those of samples A and C, which is probably caused by the thicker material of the upper sheet in the joint (DX51D of 0.9 mm).

Figs. 4 and 5 shows the obtained load–displacement curves of clinched joints and spot welded joints of the sample C. The maximum load value of clinched joint is about 1000 N and the maximum load value of spot welded joint is about 5000 N. Fig. 6 shows the obtained load–displacement curves of clinched joints of all successfully made samples A, C, and E. The curve shapes of samples A and C are very similar as well as the values of their carrying capacities.

The metallographical analysis confirmed that the area with the most significant thinning in the joint is its critical area (Fig. 7).

There occurred failures in such areas during tensile tests of samples A, C and E, and during the clinching process in samples B and D. The metallographical analysis confirmed the occurrence of cracks in the TRIP steel on the die side of the joints in the round part (Fig. 8).

Fig. 9 presents a sample C with marked areas of microhardness measurements and the measured values. The...
measurements show the changes in the clinched joint, where the highest microhardness values were measured in the critical area of the clinched joint.

4. Conclusion

The paper focused on the evaluation of clinched joints of various material combinations. Microalloyed steel HSLA H220PD, TRIP steel 40/70+Z100 MBO and DX51D+Z steel were used for the experiments.

The influence of the orientation of joined materials regarding the position of punch and die of the tool was also observed. The material combinations of TRIP 40/70 with H220PD as well as TRIP 40/70 with DX51D, where TRIP steel is oriented towards the punch, are not suitable for joining by clinching, because the joints were not successfully created. Failures occur during the clinching process in the critical areas of joints. The same material combinations where TRIP steel is oriented towards the die proved to be unsuitable for joining by clinching, even though joints were created, because there occur cracks in TRIP steel, which could negatively affect the joint, especially during dynamic load. The cracks can even decrease the corrosion resistance of the joints.

The only combination that proved to be suitable for joining by clinching was a combination of H220PD materials – sample C. The carrying capacities of these samples were sufficient and the metallographical analysis confirmed no occurrence of cracks or failures in the area of clinched joints. The carrying capacity of these joints reached about 20% of the carrying capacity of resistance spot welded joints of the same materials.

The paper was elaborated within the project Center for research of control of technical, environmental and human risks for permanent development of production and products in mechanical engineering (ITMS:26220120060).

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E. Spišák, L. Kaščák, and J. Mucha (Technical University of Košice, Faculty of Mechanical Engineering, Department of Technology and Materials, Rzeszów University of Technology, Rzeszów, Poland): Joining Materials Used in Car Body Production by Clinching

The paper dealt with the evaluation of properties of joints made by clinching. The microalloyed steel H220PD ($a_0 = 0.8$ mm), the high strength steel TRIP 40/70+Z100MBO ($a_0 = 0.77$ mm) and the drawing grade steel DX51D+Z ($a_0 = 0.9$ mm) were used for the experiments. The orientation of joined materials regarding the position of punch and die of the tool has the significant effect to carrying capacities of the joints. The TRIP steel is not suitable material for press joining method, even with both observed combination – with H220PD or DX51D. The carrying capacity of these joints was approximately 20% of the carrying capacity of resistance spot welded joints.
NANOINDENTATION TESTING OF COMPOSITE BASED ON COLLAGEN AND POLY(DL-LACTIDE) NANOFIBERS

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Keywords: nanoindentation, poly(DL-lactide), collagen, composite, nanofibers

1. Introduction

In bone tissue engineering, there is a great need to engineer multi-phase materials that combine the advantages exhibited by each component of the material, and that have a structure and composition similar to that of natural bone. In our project, we design biomimetic nanocomposite materials that promote the regeneration of defective bone tissue with the required rate of biodegradation. The proposed composition of the material imitates the real bone structure, and combines the advantages of nano fibers, aliphatic polyesters, collagen, and calcium phosphates. This study uses nanoindentation to evaluate the influence of various weight fractions of polymeric nanofibrous phase on the mechanical properties of a composite based on collagen type I matrix and poly(DL-lactide) nanofibers (PDLL). The preparation of composite test samples for nanoindentation and the structure of the samples are discussed.

2. Materials and methods

Composites based on PDLL (PURASORB PDL 05, Purac Biomaterials, the Netherlands; inherent viscosity 0.5 dl g⁻¹) nano fibers and collagen type I matrix were prepared. A poly(DL-lactide) nanofibrous omnidirectional filler (Fig. 1) was prepared by electrospinning (NS 8A 1600, Elmacro Ltd., Czech Republic) from a chloroform solution. Collagen matrix ISC₄₀ was isolated from fish skin (carp) under denaturing conditions (40 °C, acetic acid, 30 min) followed by lyophilization according to Pešáková et al. Composite samples were prepared with 6 different weight fractions of nanofibers (0, 60, 70, 73, 80, 87 wt.%) and hardened onto polymethylmethacrylate supporting plates (AZ Plastik, Czech Republic).

Briefly, all composite samples were prepared by impregnation of PDLL with collagen/deionized water dispersion. The collagenous dispersion was prepared in the IKA D118 homogenisator (IKA Werke GmbH, Germany) (at a rotation speed of 20,000 min⁻¹ for 2 minutes) by dispersion of 0.5 g of collagen in 100 g of deionized water. The weighed amount of the PDLL nanofibrous layer was placed on to separating foil and impregnated with a weighed amount of collagenous dispersion in order to achieve the chosen weight fraction of the nanofibrous filler after water evaporation (at room temperature). Four layers prepared using this procedure were cut into an appropriate size and laid on a polymethylmethacrylate supporting plate. Finally, the supporting plate and the composite were covered by a separating foil and hardened at 50 °C under a pressure of 4 kPa. A relatively low temperature was chosen to be below the glass-transition temperature of collagen and PDLL.

The assessment of various weight fractions of the nanofibrous phase on the mechanical properties (reduced elastic modulus $E_r$) of the composites was studied using the nanoindentation mode, which is an option of the Hysitron TriboIndenter™ TI 950 nanomechanical instrument (Hysitron, USA). A Berkovich diamond fluid tip with apex radius ~120 nm was used for the nanoindentation tests. For each tested composite, indents were applied on five $60 \times 60 \mu m$ areas as a matrix of 5 × 5 indents with 15 μm separation (with 25 μN applied force, lift height 100nm, preload 1 μN).

The prepared composites were also investigated by image analysis, using a QUANTA 450 electron SEM microscope (FEI Company, USA) under a high vacuum, with an Au coating film on the samples. A statistical evaluation was carried out using the following methods (STATGRAPHICS Centurion XV, StatPoint, USA): the statistically significant differences were checked by nonparametric methods (the Kruskal-
Wallis test, $\alpha = 0.05$); the Mann-Whitney test was used as a post hoc test ($\alpha = 0.05$); and the confidence intervals for the mean values were calculated at a significance level of $\alpha = 0.05$.

3. Results and discussion

The reduced elastic modulus of composites based on collagen matrix and PDLL a nanofibrous phase was measured (Fig. 2).

![Graph showing the reduced modulus $E_r$ of the tested composites](image)

Fig. 2. The reduced modulus $E_r$ of the tested composites (* denotes values without statistically significant differences, Mann-Whitney post hoc test, $\alpha = 0.05$)

The obtained value for the modulus of pure collagen matrix (0 wt.% is in reasonable agreement with earlier results obtained by various methods (2–11.5 GPa)$^6$. After additions of 60 wt.% of nanofibers, the modulus decreases markedly from 5.45–3.95 GPa to 1.17–0.91 GPa. A further decrease in the reduced modulus (in the case of composites with 70–87 wt.% of PDLL) is less marked.

It can be deduced from the trend of this decrease that the higher the amount of PDLL, the lower the reduced modulus will be. This finding can be explained by the lower elastic modulus of the PDLL precursor used for electrospinning the nanofibers (0.47–0.59 GPa)$^7$. The omnidirectional orientation of the nanofibrous phase and mainly the porosity of the composites are probably other important factors that influence the process (Fig. 3). It should be noted that the $E_r$ values can also be partly attributed to the fact that in nanoindentation the Young’s modulus represents the lateral elasticity at the surface, rather than the bulk stiffness. For further analyses in our study, it will be necessary to improve the preparation of the composite samples. The improvement will focus on a superior fibrous phase filling with collagenous matrix, and on applying the hardening process under higher pressure. The porosity of the samples will be analysed.

4. Conclusion

The results provide an assessment of the different weight fraction of the composite reinforcing phase. In general, the PDLL nanofibrous phase decreases the elastic modulus of the composites studied here. The nanoindentation method seems to be a suitable tool for determining the mechanical properties of composite materials variously modified at the nanoscale.

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REFERENCES


T. Suchý$^{a,b}$, Z. Sucharda$^a$, M. Šupová$^b$, K. Balík$^b$, J. Šepitka$^a$, and J. Lukeš$^a$ ($^a$CTU in Prague, Fac. of Mechanical Eng., $^b$Institute of Rock Structure and Mechanics, ASCR, v.v.i., Czech Republic): Nanoindentation Testing of Composite Based on Collagen and Poly(DL-Lactide) Nanofibers

The influence of various weight fractions (0, 60, 70, 73, 80 and 87 wt.%) of the poly(DL-lactide) nanofibers on the mechanical properties of the composite based on collagen type I matrix was evaluated by nanoindentation. After addition of 60 wt.% of nanofibers, the reduced elastic modulus decreases markedly (from 5.45–3.95 GPa to 1.17–0.91 GPa) while a further decrease (70–87 wt.%) is less marked.
1. Introduction

Gypsum hydration starts right after the mixing of water with gypsum. The process of hydration and setting depends on multiple factors. These effects can be observed on several scales. Usually, two levels (micro- and macro-scale) are considered at least. Material properties of hardened gypsum on macro-level (e.g. thermal and mechanical properties) depend on the material structure (namely porosity) and prevailing matrix properties. As the main factor, water to gypsum ratio determines the value of total porosity which is an effect of over-stoichiometric water. Micro-level material properties depend on microstructural parameters like chemical purity of the used gypsum (plaster); ratio between the three main components of the gypsum binder, i.e. calcium sulfate anhydrite (ν), calcium sulfate hemihydrate (β) and calcium sulfate dihydrate (α); some impurities and eventually additives; size and ordering of calcium sulfate dihydrate crystals; etc.

The main objective of this paper was to compare macro- and micro-elastic properties of studied gypsum materials and to find the dependence between macro- and micromechanical properties in connection with porosity.

2. Materials and samples

Three different materials on gypsum basis were selected for the testing. The first one was commercially available dental gypsum Interdent based on α-calcium sulfate hemihydrate (“Dental gypsum series”). The water to gypsum ratio was 0.2 in this case. The second one was a fly ash desulfurization gypsum (FGD) based on β-calcium sulfate hemihydrate produced at Electric Power Station Počerady (Czech Republic). These samples were denoted as “FGD gypsum series”. The water to gypsum ratio was 0.627 in this case. The last series was commercially produced grey gypsum based on β-calcium sulfate hemihydrate (Gypstrend Ltd. – Kobořice near Opava in Czech Republic) denoted as grey gypsum (after typical color of this gypsum which contained 50 % of natural calcium sulfate dihydrate). The water to gypsum ratio was 0.71. Table I shows the basic material properties as bulk density, total porosity and values of micro-porosity lower then 1 μm. This part of the porosity was assumed to be naturally included in the nanoindentation data since the indentation volume under the tip covered a region of ~1–2 μm³.

Table I

<table>
<thead>
<tr>
<th>Material</th>
<th>Bulk density [kg m⁻³]</th>
<th>Total open porosity [m³ m⁻³]</th>
<th>Open porosity lower then 1 μm [m³ m⁻³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dental gypsum</td>
<td>2020</td>
<td>0.19</td>
<td>0.12</td>
</tr>
<tr>
<td>FGD gypsum</td>
<td>1220</td>
<td>0.51</td>
<td>0.14</td>
</tr>
<tr>
<td>Grey gypsum</td>
<td>980</td>
<td>0.61</td>
<td>0.13</td>
</tr>
</tbody>
</table>

3. Experimental methods and results

First, the macroscopic values of dynamic Young’s modulus on macro-level were measured by non-destructive impulse excitation method which is based on measuring the fundamental resonant frequencies. The test arrangement was done for longitudinal vibration. The specimen with dimensions of 40×40×160 mm was supported in the midspan, i.e. the fundamental longitudinal nodal position. The acceleration transducer Bruel&Kjaer of Type 4513B was placed at the centre of one sample end face. The opposite end face was hit by the impact hammer Bruel&Kjaer, Type 8206. From the obtained results, the weight of the sample and the dimension of the sample, values of dynamic Young’s modulus were calculated. Total open porosity and values of micro-porosity were calculated from results obtained from mercury porosimetry and pycnometric density measurements.

Micromechanical properties of dental gypsum samples were measured by using CSM Nanohardness tester. Quasi-static loading consisted of 10 s of linear loading (rate 30 mN min⁻¹), 10 s of holding period at constant peak force 5 mN and 10 s of unloading (rate 30 mN min⁻¹). The distance between individual indents was set 15 μm to avoid mutual influences. Elastic constants were evaluated for individual indents by standard Oliver and Pharr methodology. Poisson’s ratio was estimated to be 0.2 for all cases. Grid nanoindentation and deconvolution techniques were applied. Three phase microstructural system was assumed based on the shape of experimental histograms of elastic moduli. Thus, the anisotropy of gypsum...
crystals was replaced by the phase differences at a deconvolution process. The dental gypsum composed of one dominant phase (E=47.2 GPa, 71.2 %) and two minor phases (E=19.4 GPa, 4.4 % and E=56.3 GPa, 24.4 %). To compare results from different methods, macroscopic elastic properties were predicted from analytical homogenization scheme, namely Mori-Tanaka method, with the assumption of a two-phase composite – matrix (having the properties received from nanoindentation and lower level homogenization) of the three phase system and air pores larger than 1 μm. The homogenized Young’s modulus for the matrix was 34.8 GPa. Results from macroscopic dynamic measurements showed on the value 36 GPa, i.e. the agreement of the macroscopic value and the micromechanically predicted one was within 3.3 % in this case.

Since the direct micromechanical measurements on FGD and grey gypsum samples faced significant obstacles in the form of very high porosity and roughness it was decided to use inverse analysis and predict micromechanical properties of the matrix from the macroscopic ones using the same principles as in case of dental gypsum. The obtained results are summarized in Table II. It can be seen that macro elastic properties of FGD and grey gypsum samples (based on β-gypsum composition) are approximately 2.6–2.8× lower then those for dental gypsum (α-gypsum composition).

<table>
<thead>
<tr>
<th>Gypsum</th>
<th>Macroscopic Young’s modulus [GPa]</th>
<th>Microscopic (homogenized matrix) Young’s modulus [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dental</td>
<td>36/34.8</td>
<td>40.0</td>
</tr>
<tr>
<td>FGD</td>
<td>7/</td>
<td>15.2*</td>
</tr>
<tr>
<td>Grey</td>
<td>5/</td>
<td>14.2*</td>
</tr>
</tbody>
</table>

Note: * denotes results obtained from inverse analysis. An arrow indicates the analysis direction.

4. Conclusions

The paper presents comparison of micro- and macro-mechanical properties of several types of gypsum materials in dependence on their different chemical origin and porosity. Grid nanoindentation, statistical deconvolution and porosimetry were utilized. Good agreement within 3.3 % was found in case of dental gypsum samples based on α-gypsum composition. The analytical homogenization was used in the prediction of either microscopic matrix properties (for dental gypsum) or for the inverse analysis of microscopic properties from the known macroscopic ones (for FGD and grey gypsum samples). This analysis points on the approximately 2.6–2.8× lower microscopic elastic properties of β-gypsum based samples. Such hypothesis will be subsequently verified using nanoindentation which was beyond the scope of this contribution at present.

Support of the Czech Science Foundation (P105/12/0824) and Ministry of Education of the Czech Republic (MSM 6840770003) is gratefully acknowledged.

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P. Tesárek, T. Plachý, P. Ryparová, and J. Němeček (Czech Technical University in Prague, Faculty of Civil Engineering): Micromechanical Properties of Different Materials on Gypsum Basis

Micro- and macro-level elastic properties of three types of gypsum samples (dental gypsum, flue gas desulphurization gypsum and grey gypsum) were compared. Grid nanoindentation, statistical deconvolution and porosimetry were used on lower composite level and non-destructive impulse method on macro-scale. The transition between the scales was computationally maintained by the Mori-Tanaka homogenization method. Good agreement was achieved between experiments and numerical prediction.

Obtained results also showed that the micro-elastic properties predicted (by inverse analysis) for FGD and grey gypsum samples (β-gypsum composition) are approximately 2.6–2.8× lower then those for dental gypsum (α-gypsum composition).
IMPACT OF BLEACHING GELS ON DENTAL ENAMEL MICROHARDNESS AND 3D SURFACE ROUGHNESS

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Keywords: enamel, bleaching, microhardness, 3D surface roughness

1. Introduction

At this time, the popularity of dental bleaching has increased in esthetic dentistry. Tooth bleaching using different oxidizing agents as hydrogen peroxide or carbamide peroxide is one of the most spread procedures\textsuperscript{1–4}. The effect of bleaching treatment on dental enamel has long been a concern of many dentists, because bleaching agents react with enamel and cause chemical, structural and mechanical changes on the enamel surfaces. One of the most common methods of evaluation of changes in surface topography\textsuperscript{5,6} is electron microscopy, stylus profilometry or atomic force microscopy\textsuperscript{7}. The aim of this work is to investigate the effect of two different tooth bleaching gels containing 22 % carbamide peroxide and 38 % hydrogen peroxide on surface microhardness and properties of dental enamel by the 3D surface topography.

2. Materials and experimental methods

Four extracted teeth human third molars were used for experiment. Enamel slabs of 5.0x5.0x1.5 mm were cut from the buccal and lingual surfaces by using a precision slow speed diamond saw with water cooling. Each cut slab was embedded in dentacryl. Enamel surface was wet grounded to achieve flat surface by using 400, 600, 1000 and 2500 grit silicon carbide papers. Then the prepared slabs were placed in an ultrasonic cleaner for 3 min. Samples were stored in distilled water at room temperature prior to the experiment. A total of 8 enamel specimens were prepared for the experiments.

Two different bleaching gels were applied on the prepared specimens. For experiments, „in home“ Yotuel\textsuperscript{®} Patient with 22 % carbamide peroxide (Biocosmetics Laboratories) and „in office“ Opalescence Boost with 38 % hydrogen peroxide (Ultradent products) were used.

Yotuel\textsuperscript{®} Patient was applied in 1 cycle for 3 hours and Opalescence Boost was applied in 4 cycles for 15 minutes. After bleaching treatment samples were cleaned in distilled water and Baseline microhardness was measured before treatment with a microhardness Vickers indenter (LECO LM 247 AT) at a 100 g load and 12 s dwell time. The mean Vickers hardness numbers (VHN) were derived from five indentations made across the enamel surface of each specimen.

After microhardness testing, surface topography of the specimens was evaluated on a 3D surface area by the Talysurf CLI 1000 device with non contact confocal gauge. The topography data were visualized and evaluated using commercial software Talymap Platinum. Measured area was 1.25x1.25 mm. The filter cut off which separates the roughness and waviness area \( \lambda_c \) was set to 0.25x0.25 mm. These roughness parameters were measured: the core roughness depth \( S_k \) – the height difference between intersection points of the found least mean square line, the reduced summit height \( S_{pk} \) – the height of the upper left triangle and the reduced valley depth \( S_{vk} \) – the height of the triangle drawn at 100 %.

3. Results

The confocal microscopy image shows the bleached surface in Fig. 1. The measured values of microhardness of showed enamel before and after treatment remain almost the same (see Fig. 2).

The area measured by the Talysurf CLI 1000 device is shown in Fig. 3. The values of selected parameters before and after bleaching treatment are shown in Tab. I. The results show that bleaching gel Yotuel\textsuperscript{®} Patient has a bigger impact on surface roughness parameters than Opalescence Boost PF. The results from amplitude distribution and material ratio curves (Fig. 4) confirmed that bleaching gels have effects on topography of enamel. After treat-

![Fig. 1. The bleached surface of dental enamel in a confocal micrograph](image-url)
4. Conclusion

The obtained results show that bleaching treatment has only small effects on surface microhardness of dental enamel. The values remained the same. All measured parameters of surface roughness $S_k$, $S_{pk}$, $S_{vk}$ after treatment was changed. The biggest change of surface topography was observed for Yotuel® Patient. In this case, the parameter $S_k$ decreased from 0.426 $\mu$m to 0.213 $\mu$m. The results show that “in home” bleaching gel with 22 % carbamide peroxide has bigger impact on dental enamel roughness than “in office” bleaching gel with 38 % hydrogen peroxide.

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Q. D. Tran*, K. Mañas*, E. Svoboda*, M. Bumbáček*, and Z. Joska* (*Department of Mechanical Engineering, University of Defence, ‡Department of Dental Care, Faculty of Medicine, Masaryk University): Impact of Bleaching Gels on Dental Enamel Microhardness and 3D Surface Roughness

The aim of this study was to characterize the effect of two different bleaching gels (with 22 % carbamide peroxide and with 35 % hydrogen peroxide) on the surface roughness and microhardness of enamel. Bleaching gels were applied according to manufacturer’s instructions. For characterization of surface microhardness and 3D surface roughness, non contact 3D profilimeter TalySurf CLI 1000 and Vickers microhardness method were used. The results of surface microhardness did not show significant changes from baseline for both gels. 3D surface roughness parameters showed that “in home” gel with 22 % carbamide peroxide caused bigger changes of enamel topography than “in office” gel with 35 % hydrogen peroxide.

Table I

<table>
<thead>
<tr>
<th>Surface roughness parameter</th>
<th>Yotuel® Patient</th>
<th>Opalescence Boost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method of treatment</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grounded</td>
<td>Bleached</td>
<td>Grounded</td>
</tr>
<tr>
<td>$S_{pk}$</td>
<td>0.416</td>
<td>0.626</td>
</tr>
<tr>
<td>$S_k$</td>
<td>0.426</td>
<td>0.213</td>
</tr>
<tr>
<td>$S_{vk}$</td>
<td>0.137</td>
<td>0.254</td>
</tr>
</tbody>
</table>