



Digital Image Correlation and nanoindentation in evaluation of material parameters of cancellous bone microstructure

G. Kokot ^{a,*}, K. Skalski ^b, A. Makuch ^b, W. Ogierman ^a

^a Institute of Computational Mechanics and Engineering, Silesian University of Technology,
ul. Konarskiego 18a, 44-100 Gliwice, Poland

^b Institute of Precision Mechanics, ul. Duchnicka 3, 01-796, Warszawa, Poland

* Corresponding e-mail address: groot@amme.com

ABSTRACT

Purpose: Purpose of this paper is to present the possibilities of the application of the two methods: Digital Image Correlation and nanoindentation in porous bone tissues testing. Firstly, as a tool in the evaluation process of material parameters for porous microstructures, such as bone tissues or other foams and, secondly, as validation and verification tools for finite element analysis of bone or foams structures. Those methods are helpful when the high accuracy of the mechanical parameters of porous microstructures is required.

Design/methodology/approach: Two methods: Digital Image Correlation (DIC) and nanoindentation are used as an efficient approach in the evaluation process of material parameters or constitutive relationship of porous structures like bone tissues. Digital image correlation enlarges the accuracy of classical mechanical tests and the nanoindentation allows to look inside the microstructure.

Findings: The proposed methods were found to be effective in experimental testing and material parameters evaluation process of some special materials. Among them are porous structures, such as bone tissue. Additionally, the DIC is an excellent tool for finite element model validation and results verification.

Practical implications: The presented method based on the combination of the Digital Image Correlation and nanoindentation presents new possibilities in material testing fields, material behavior and parameters evaluation. They have great advantages, among others, in the field of testing of porous bone structure or determining the mechanical parameters of bone tissue.

Originality/value: The paper presents methods for testing the complicated porous bone structures: evaluating mechanical behavior of the whole structure and evaluating mechanical properties of the single element of the structure. The mechanical parameters of human cancellous bone structures are presented as the preliminary research results.

Keywords: Digital Image Correlation; Nanoindentation; Porous structure; Bone tissue; Experimental testing

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METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

The mechanical parameters of the porous cancellous bone structures can be evaluated in two ways. The first one is to obtain the mechanical parameters of the structure built of hundreds small trabeculae, and the second one is to obtain the mechanical parameters of the single trabeculae. This is connected with the hierarchical structure of the bone [1,2].

Most of the mechanical parameters of bone tissues presented in the literature were determined on the structural level using classical experimental method taken directly from mechanics [1,3]. These are mostly tension/compression test and three or four bending tests. However, while using those methods, we often encounter difficulties which are mostly connected with displacement measurement and low precision and accuracy of results. This is due to small specimen size, complicated mounting way and porous structure of the tested material, often additionally under wet condition. The other problem is the hierarchical structure of the bone which strongly influences the mechanical properties on the macro- and micro level [1,4]. To avoid these technical problems the combination of classical research methods of mechanical properties of bone tissues with contemporary optical methods of displacement measurement is proposed. Such combinations considerably enhance the possibility of evaluating the mechanical properties of bone tissues, particularly taking into account the porous microstructure of the bone tissue. In the presented paper the digital image correlation system is used as the optical displacement and strain measurement system. The combination of this method with classical compression test was successfully used in the prediction of Young's modulus of trabeculae structure in microscale using macro-scale's relationships between bone density and mechanical properties [5]. This seems to be an efficient way for testing the porous bone microstructures, where the geometry layout influences the mechanical behavior. In the cases where the exact mechanical parameter of single trabeculae of the microstructures is needed to be known the nanoindentation method is proposed. It allows to discover parameters even on the nanolevel of the inner structure of the specimen.

2. Materials and methods

2.1. Digital Image Correlation (DIC)

The digital image correlation is an optical measuring instrument for true full field, non-contact and three-dimensional analysis of displacements and strains on

components and specimens. This method is characterized by a high measuring resolution and real time non-contact measurement. The non-contact analysis of displacements makes it the excellent choice in the case of the porous materials like bone tissues.

Digital image correlation as a tool for surface deformation measurements has found widespread use and acceptance in the field of experimental mechanics. The method is known to reconstruct displacements with subpixel accuracy and tangential surface strains in the micro strain range.

The digital image correlation is the method where the random speckle pattern applied on the specimen surface is observed by two or three cameras during the loading phase. The correlation algorithm compares gathered images, transforms correspondingly marked positions in cameras into 3D coordinates and correlates each object points movement in three dimension. As a result of this correlation the full field colorful displacement and strain maps are evaluated. The measuring range of the DIC (Fig. 1) is complementary to the Electronic Speckle Pattern Interferometry but a DIC measurement is free of many disadvantages connected with ESPI. It is simple to use and cost effective.

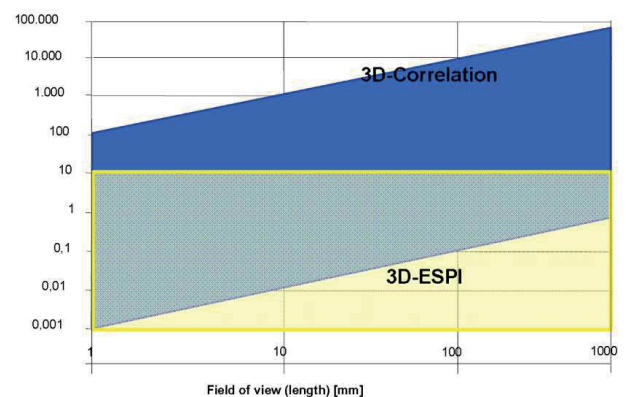


Fig. 1. The measurement range of the DIC system

The applied optical methods or strain gauges for displacement or strain measuring in the porous bone tissues allow for measurements only in the specified points of the structure. There is no wider information about the methods for accurate analysis of the local deformation, especially on the level of microstructure where the information is given in the form of the strain field. Here, the digital image correlation can be helpful. In connection with the classical testing machine, it is an excellent tool for classical experiments like tension/compression test conducted on the porous structure where the classical methods of the strain

measurements fail. The classical testing stand assisted with DIC system is presented in Figure 2.



Fig. 2. The testing stand with the Digital Image Correlation system

The basis of the measuring methodology is the speckle pattern on the tested surface. This is a small disadvantage of this method - covering the surface of the measured object with special speckle pattern (Fig. 3) taking into account the bone structure as the tested material.

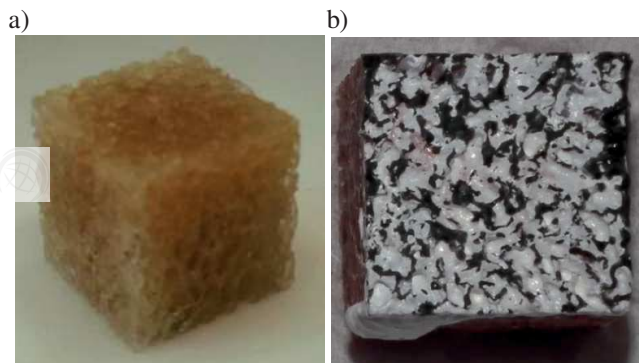
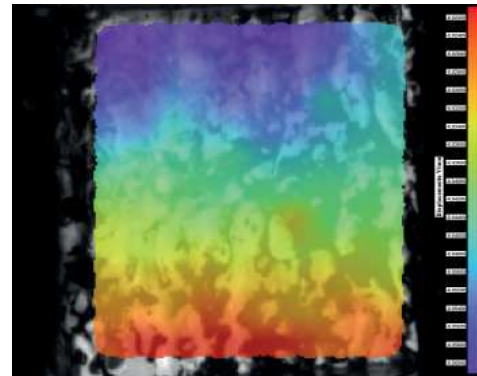


Fig. 3. The cancellous bone specimen without (a) and with special speckle pattern (b)

One of the main advantages of this method is achieving the results on the whole specimen surface as the colorful maps comparable to maps of results of the finite element analysis. Additionally, they can be presented directly on the tested surface or as a separate colorful map (Fig. 4). It allows directly to validate and verify the numerical model and numerical results obtained using the finite element method (Fig. 5) [6].

a)



b)

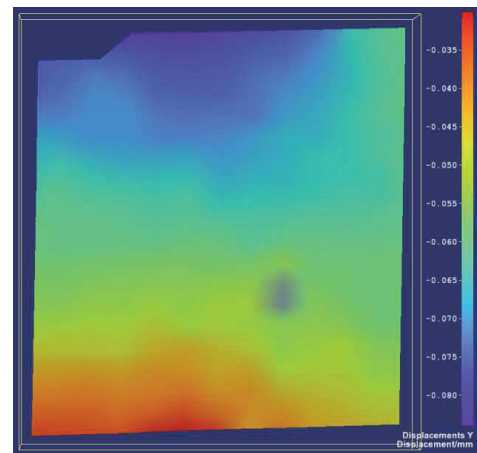


Fig. 4. The colorful displacement map of the testing sample (a) presented on the tested surface and as a separate colorful map (b), maps present the displacement filed in chosen direction

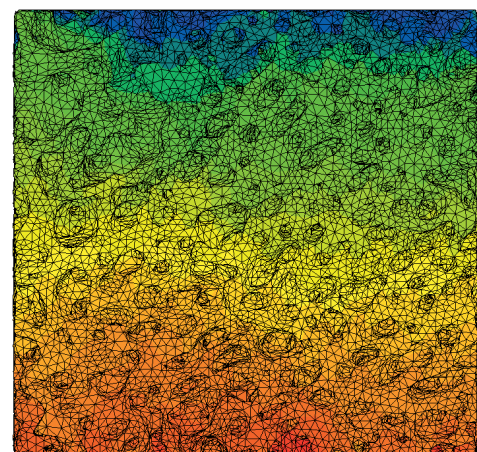


Fig. 5. The colorful displacement map as the result of the finite element simulation of the compression test of the mechanically tested sample

The examples of the compression tests results, where displacements and strain under applied load were measured using the digital image correlation method in the form of the stress-strain curves are presented in Figure 6. Compression tests of the trabecular bone structure were conducted using the cubic shaped cancellous bone samples with dimension 10x10x10 mm (Fig. 3) with the test speed 0.4 mm/min using standard MTS Insigh testing machine with load capacity of the 2 kN. The obtained stress-strain curves show the large linear elastic range (Fig. 6). This is the important information from the finite element analysis point of view. It means that in typical numerical simulation bone can be modeled as the linear elastic material, what simplified the process of material model definition. Of course, in complicated analysis e.g. bone remodeling, the much more complicated material models describing full bone structure behavior with time dependence and bone orthotropy should be taken into account.

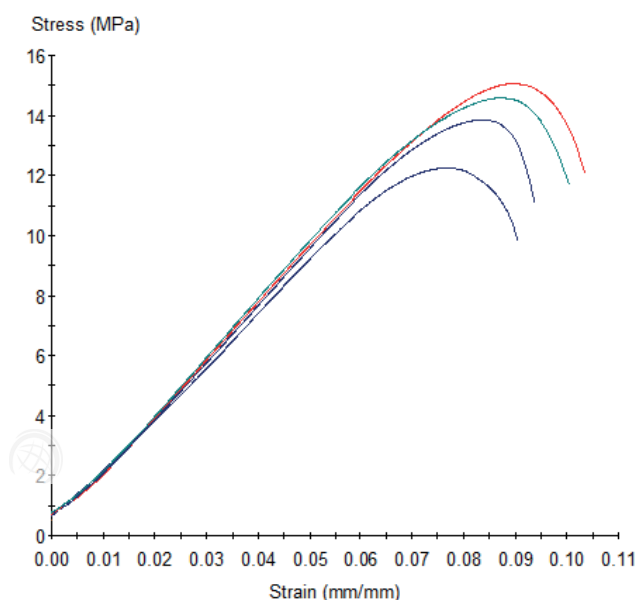


Fig. 6. The stress-strain curve for compression test of cancellous bone structure

The structural Young modulus calculated on the base of presented stress-strain curves vary from 0.85-1.14 GPa. The detailed results can be found in [6].

2.2. Nanoindentation

Nanoindentation is increasingly widely used in biomechanics. It comes from the experimental mechanics

for testing the hardness and Young modulus of thin coatings. The main advantage is the measurement on the micro - or even nanostructure level [7,8].

This method is characterized by its penetration force range: from mili- to microniutons, making it applicable in fields where other methods fail. This method has also found successful application in bone testing, due to the described advantages [8,9]. The main idea for testing is that the penetrator penetrates the sample with the specified force and then control the sample while unloading. The first part of the unloading curve relates to the ideal elastic behavior of the sample. The results of the nanoindentation test are presented as force penetration P_m – penetration depth h_m curve (Fig. 7).

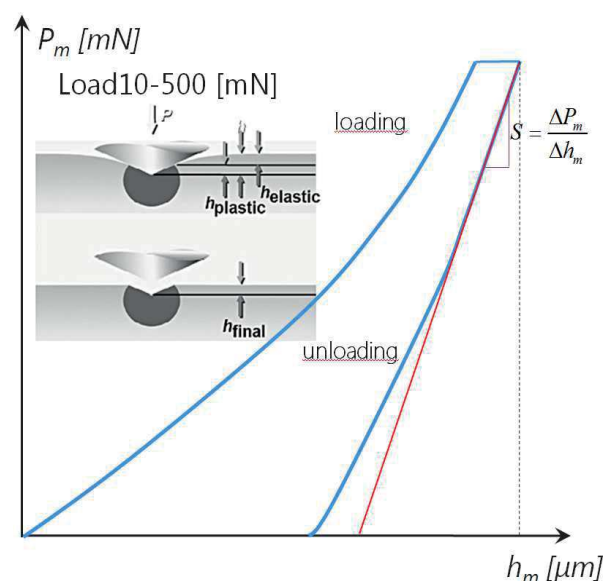


Fig. 7. The nanoindentation P_m - h_m curve

As mechanical material properties two basic parameters are evaluated: hardness H_{IT} and Young modulus E_{IT} . The hardness is calculated as maximum force P_m divided by projected contact area A_p :

$$H_{IT} = \frac{P_m}{A_p} \quad (1)$$

The most popular method of the Young modulus determination is the Olivier and Pharr power law method [10]. The power law method recognizes the fact that the first portion of the unloading curve may not be linear, and can be described by a simple power law relationship where Young modulus is calculated as follows:

$$E_{IT} = \frac{(1-\nu^2)}{\frac{1}{E_{IT}^*} - \frac{(1-\nu_i^2)}{E_i}} \quad (2)$$

where:

E_{IT}^* – reduced modulus of the indentation contact,

ν – Poisson's ratio of the sample,

ν_i – Poisson's ratio of the indenter,

P_m – loading force,

h_m – penetration depth (Fig. 8).

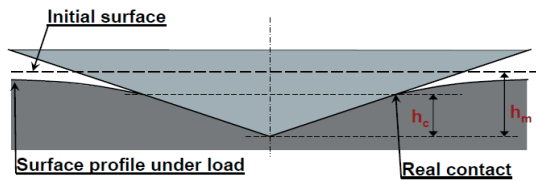


Fig. 8. The representation of the indentation process – sample contact

The nanoindentation is basically used for determination of the force-depth curve and evaluation of the standard material property as the Young modulus and microhardness. The other measured values helpful from, for example, constitutive equation formulation point of view in the time dependent domain [11], are indentation creep (C_{IT}), indentation relaxation (R_{IT}), elastic and plastic part of the indentation work (W_{elast} , W_{plast}) (Fig. 9), a phase angle φ between the force and displacement signals in dynamic mechanical analysis which characterizes the viscous character of the material. The important parameters are maximum force, testing velocity and holding time. The holding time is used for determining the effect of viscoelastic properties of the specimen.

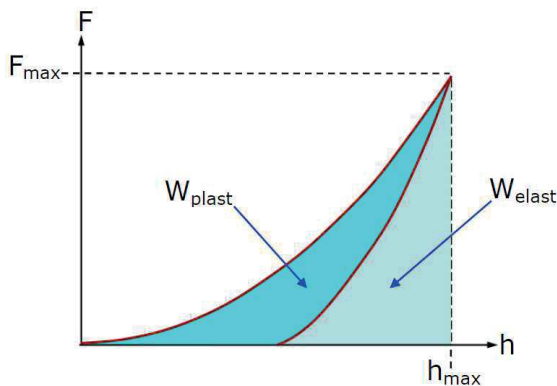
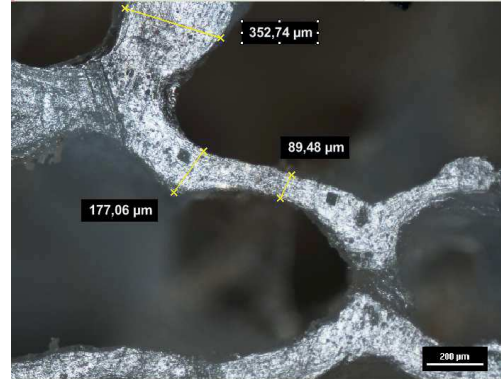


Fig. 9. The work definition in indentation

The preliminary nanoindentation test of human cancellous bone has been done using the CSM Microhardness Tester. In Figure 10, a sample of nanoindentation test is presented. The pictures present microphotography of the testing places, where the single trabeculae are visible with their dimensions. The zoomed part with the Vickers indenter indentation points is presented in Figure 10 b.

a)



b)

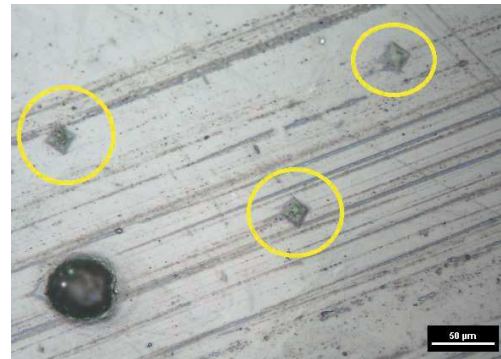


Fig. 10. The tested sample (a) with Vickers indentation points (b)

The achieved experimental results are presented in Figure 11, in the form of a set of nanoindentation curves. The calculated values of the microhardness according to formula (1) and Young modulus calculated according to formula (2) are presented in Table 1 and Table 2.

The results of measurements of microhardness and elasticity of the trabecular bone (Table 1 and Table 2) were obtained with the following tests parameters: velocity (v) – 600 mN/min, maximum loading/unloading force (P_{max}) 500 mN and hold time (τ) – 0.1 s and 1000 s, respectively. It was observed that the process parameters can significantly affect the mechanical properties of bone structures at a microscopic level [14].

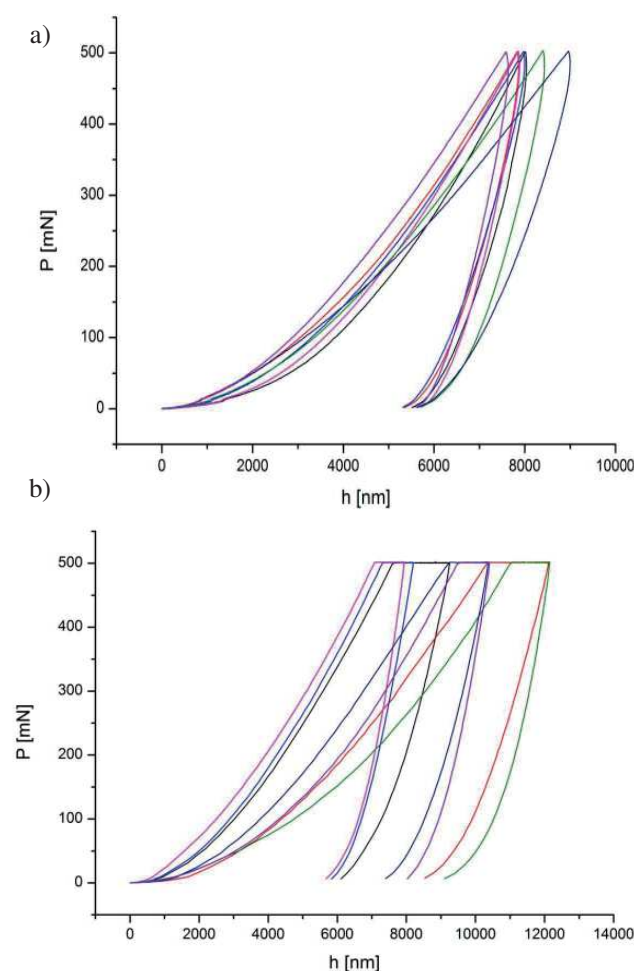


Fig. 11. The nanoindentation curves for tested sample: $\tau=0.1$ s (a), $\tau=1000$ s (b)

The nanoindentation curves were obtained for two holding times : $\tau=0.1$ s ; $\tau=1000$ s.

Table 1.
Nanoindentation results for a cancellous tissue $\tau=0.1$ s

Test	Microhardness – H_{IT} ,	Young modulus – E_{IT} ,
	MPa	GPa
1	415.73	8.04
2	442.88	8.03
3	452.89	6.87
4	426.55	8.80
5	377.89	7.39
6	360.54	5.34
7	455.76	9.45

Table 2.

Nanoindentation results for a cancellous tissue $\tau=1000$ s

Test	Microhardness – H_{IT} ,	Young modulus – E_{IT} ,
	MPa	GPa
1	319.91	5.65
2	181.66	3.61
3	395.11	7.85
4	402.42	9.65
5	162.5	5.35
6	243.98	5.14
7	228.06	6.35

3. Conclusions

The application of classical methods of experimental research in the evaluation of mechanical properties of bone tissues, in most cases, allows to determine the material property only on the macrostructure level and often it is characterized by a low precision of measurement. It is also difficult to evaluate accurate stress-strain characteristics required in numerical modeling of complicated phenomena, such as bone remodeling or fracture. In many cases, it is difficult to perform precise displacement measurements. We are still looking for new methods for more precise and accurate evaluation of bone tissues mechanical properties, with particular consideration of the hierarchical structure of the bone.

The paper presents preliminary research results in the field of the evaluation of mechanical properties of bone tissues using new measurement techniques in experimental testing of porous structure of bones, such as Digital Image Correlation (DIC) and nanoindentation. Those methods are successfully applied in mechanics and applying them in the biomechanics is a promising research direction. The preliminary research using nanoindentation in the evaluation of individual material properties of single trabeculae, osteons or lamellae has been conducted and the achieved results are very promising. The presented methods can be helpful in the much more complicated evaluation process of constitutive equations describing in detail bone structure mechanical behavior.

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References

- [1] J.Y. Rho, L. Kuhn-Spearing, P. Zioupos, Mechanical properties and the hierarchical structure of bone, *Medical Engineering & Physics* 20 (1998) 92-102.
- [2] R.B. Ashman, J.Y. Rho, Elastic modulus of trabecular bone material. *Journal of Biomechanics* 21 (1988) 77-81.
- [3] Y.H. An, R.A. Draughn, Mechanical testing of bone and the bone-implant interface. CRC Press, Boca Raton, USA, 2000.
- [4] M. Demiral M., A. Abdel-Wahab, V. Silberschmidt, A numerical study on indentation properties of cortical bone tissue, Influence of anisotropy, *Acta of Bioengineering and Biomechanics* 17/2 (2015) 3-14.
- [5] Ł. Cyganik., M. Binkowski, G. Kokot, T. Rusin, P. Popik, F. Bolechala., R. Nowak, Z. Wróbel, A John, Prediction of Young's modulus of trabeculae in microscale using macro-scale's relationships between bone density and mechanical properties. *Journal of the Mechanical Behavior of Biomedical Materials* 36, (2014) 120-134.
- [6] G. Kokot, evaluation of bone tissues mechanical properties using digital image correlation, nano-indentation and numerical simulations, Publishing of Silesian University of Technology, 2013 (in Polish).
- [7] J. Nemecek: Nanoindentation in material science, Intech, 2012.
- [8] C.E. Hoffler, X.E. Guo, P.K. Zysset, S.A. Goldstein, An application of nanoindentation technique to measure bone tissue lamellae properties. *Journal of Biomechanics Engineering* 127/7 (2005) 1046-1053.
- [9] G. Kokot, M. Binkowski, A. John, B. Gzik-Zroska: Advanced mechanical testing methods in determining bone material parameters. *Mechanika, Proceedings of 17th International Conference*, Kaunas, 2012, 139-143.
- [10] W.C. Olivier, G.M. Pharr, An improved technique for determining hardness and elastic-modulus using load and displacement sensing indentation experiments, *Journal of Materials Research* 7/6 (1992) 1564-1583.
- [11] M. Pawlikowski, K. Barcz, Non-linear viscoelastic constitutive model for bovine cortical bone tissue, *Biocybernetics and biomedical engineering* 36 (2016) 491-498.
- [12] D. Christen, A. Levchuk, S. Schor, P. Schneider, S.K. Boyd, A. Müller, Deformable image registration and 3D strain mapping for the quantitative assessment of cortical bone microdamage, *Journal of the Mechanical Behavior of Biomedical Materials* 8 (2012) 184-193.
- [13] P. Sztetek M. Vanleene, R. Olsson, R. Collinson, A.A. Pitsillides, S. Shefelbine, Using digital image correlation to determine bone surface strains during loading and after adaptation of the mouse tibia, *Journal of Biomechanics* 43 (2010) 599-605.
- [14] A. Makuch, K. Skalski, M. Pawlikowski, The influence of the cumulated deformation energy in the measurement by the DSI method on the selected mechanical properties of bone tissues, *Acta of Bioengineering and Biomechanics* (2016).

