Key Engineering Materials Vol. 606 (2014) pp 133-136
© (2014) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/KEM.606.133

Nanoindentation of Human Tooth Dentin

Aleš Jíra[1,a], Jiří Němeček[1,b]

[1]Czech Technical University in Prague, Faculty of Civil Engineering, Thákurova 7, 166 29 Prague, Czech Republic

[a]jira@fsv.cvut.cz, [b]jiri.nemecek@fsv.cvut.cz

Keywords: Dentin, nanoindentation, elastic modulus, anisotropy.

Abstract. Biological tissues belong to the group of materials highly dependent on its micro structure. A variety of results may be found in the study of human teeth whose local composition and mechanical characteristics show considerable variations. The article deals with the measurement of elastic micromechanical characteristics in the root part of human tooth dentin. An extracted tooth was analyzed in two typical directions, i.e. longitudinal (from the tooth crown-neck to the root) and transverse (in the middle of the root part). Values of the modulus of elasticity in walls of the tooth root in the longitudinal and transverse directions were identified by means of nanoindentation. The development of the modulus of elasticity was measured in several parallel rows proceeding from the outer edge of the tooth root towards the root canal. The values measured in the dentin part of the tooth root ranged around ~17 GPa (cement-dentin boundary), ~23 GPa (middle part of dentin) and ~14 GPa (dentin-root part interface). In the longitudinal section, the value of the modulus of elasticity was almost constant in the majority of tested volume (~18 GPa) with some deviations in peripheral part (end of the root) due to changes in microstructural orientation from sagittal plane. The differences revealed a low degree of anisotropy of dentin.

Introduction

The knowledge of mechanical characteristics of hard dental tissues is important for understanding of the wide range of dental processes, and essential in the design of dental implants and their intraosseal parts. The study addresses micromechanical characteristics of dentin in the root of a premolar tooth. Dentin is a heterogeneous material primarily consisting of a tubular (porous), peritubular (dense tubular rim) and intertubular (the main component of the dentin wall) areas. The diameter of the tubules is 1-3 μm and its density is ~15000 tubules per mm^2 [1], see Fig. 1a.

Methodology

Nanoindentation allows measurements of highly calcified peritubular and intertubular dentin with their local variations which demonstrate the dependence of dentin mechanical behavior on its microstructure. To identify the values of modulus of elasticity, a premolar tooth extracted for orthodontic reasons was tested in two typical directions: vertically (on transverse sections) and longitudinally (on horizontal sections), as illustrated in Fig. 1b. The specimen was firstly fixed in an epoxy resin and cut in the required sections. Then it was carefully grinded, polished and cleaned in an ultrasonic bath and alcohol. Prepared surfaces were tested with CSM Instruments Nano Hardness Tester equipped with the Berkovich indenter.

To study the dependence of the indent size and the effect of tubules presence on the values of the modulus of elasticity, cyclic indentations to varying depths were conducted in the middle part of the dentin wall. A rectangular matrix containing 16 indents with mutual offset 0.04 mm was performed using force controlled test. Five cycles with the maximum loads of 10, 20, 30, 40 and 50 mN and a loading/unloading rate of 120 mN/min were prescribed. It was discovered that for relatively large indents (~750-1650 nm in depth) the measurement was not significantly affected by the porosity (presence of tubules) of the dentin as indicated by small variations in elastic moduli (24.5±1 GPa) in the given depth range. It was, therefore, assumed that using such penetration depths is suitable for further profiling of elastic properties across larger areas.

To obtain profiles of mechanical properties, indentation in the longitudinal direction was made in matrices containing 4x31 indents (vestibulo-oral direction, labeled as A in Fig. 2b) and matrices with 47x4 indents (disto-mesial direction, labeled as B in Fig. 2b). The matrices covered the tooth cross section through cement, cement-dentin boundary and inner dentin finally approaching to the root canal (Fig. 1b, 2b). The distance between individual indents was 0.04 mm. Indentation was carried out by controlled force with the maximum magnitude of 30 mN and a loading/unloading velocity of 360 mN/min. The penetration depth depended on the position in the dentin wall and ranged between ~1400-1700 nm (Fig. 2a).

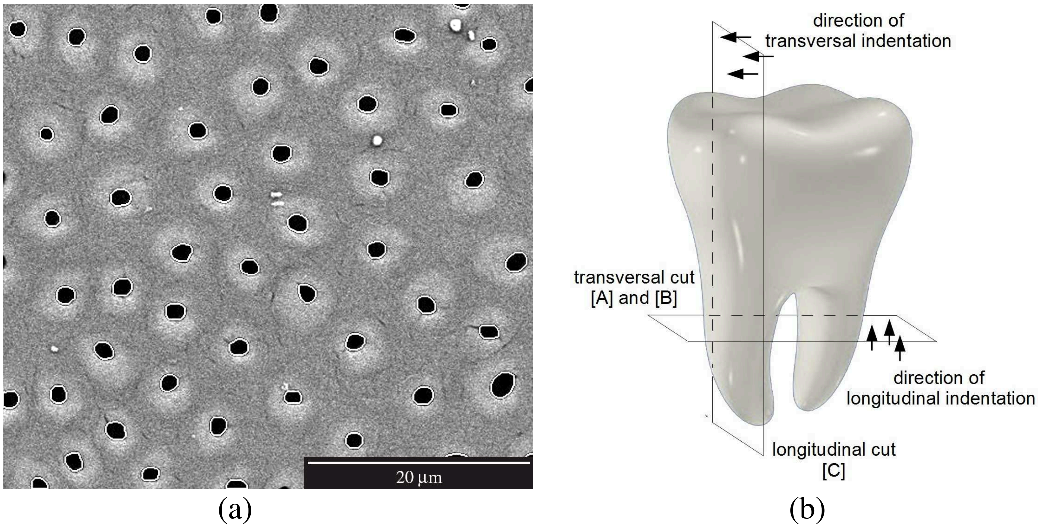

Fig. 1 (a) SEM image of tubules in dentin. (b) Schematics of tested locations and directions.

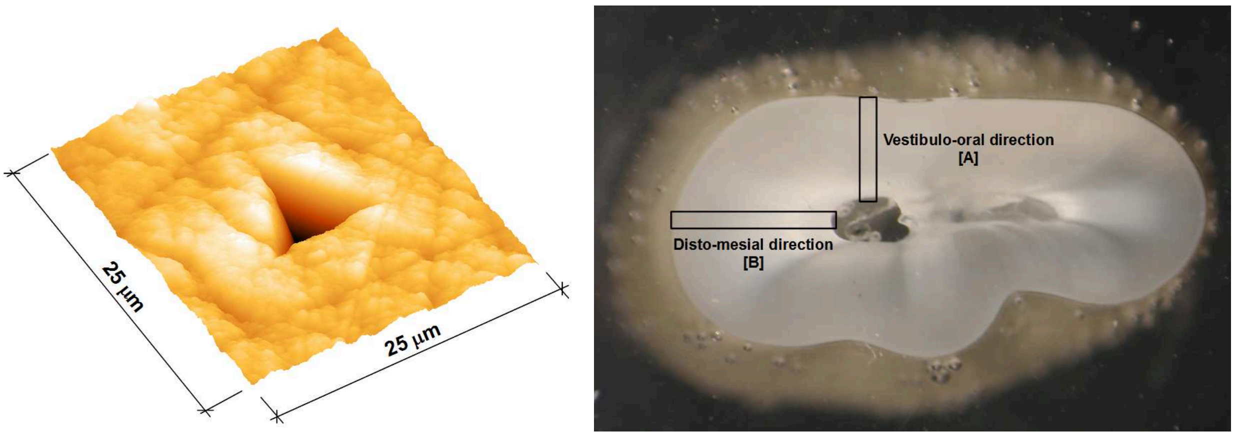

Fig. 2 (a) AFM image of an indent in the middle of the dentin and (b) transversal cut with locations of indentation matrices A and B.

Similarly, indentation in the transverse direction was performed using elongated matrices containing 190x3 indents (in the apical direction, labeled as C in Fig. 1b) with the mutual separation of indents 0.06 mm. The indentation was carried out by controlled force with the maximum magnitude of 20 mN and a loading/unloading rate of 240 mN/min. The penetration depth ranged in ~ 900-1300 nm. The matrices were located in the sagittal plane (i.e. longitudinal cut). The majority of tubules is approximately perpendicular to the sagittal plane in the majority of the tooth root. But note that the orientation of tubules deviates from the sagittal plane in the peripheral part of the root significantly. This deviation must be taken into account when evaluating elastic properties.

All measurement were evaluated with the assumption of elastic isotropic material and the Oliver-Pharr methodology [3]. The assumption on isotropy is not completely fulfilled for the dentin. However, the used evaluation can serve as a good indicator of the directional dependence of the modulus and for comparison of individual locations.

Results

The measurement of elastic modulus on the transversal cut (i.e. in the vestibulo-oral direction A) show that lower values are obtained on the outer edge of a premolar tooth (in the cement and cement-dentin boundary) ranging around ~17 GPa, and also at the dentin-root canal interface (~14 GPa). The highest values were measured in the central part of dentin reaching ~23 GPa (Fig. 3). In the disto-mesial direction (B) on the same cut, the development of the modulus of elasticity is analogous with the (A) direction. The lowest value of the modulus of elasticity in the disto-mesial direction was measured at the interface of dentin and the root canal (~13 GPa) and in dentin on the boundary with cement (~17 GPa). The highest value was measured in the central part of dentin (~24 GPa).

The changes in elastic moduli could be partly attributed to anisotropy of the dentin in different parts [4]. However, more likely, the decrease in elastic modulus is caused by the decrease in the material density. This situation can be seen in microscopic images (Fig. 1, 2) where the density of tubules changes towards outer dentin parts. The tubules are wider in the direction towards the pulp. Note, that the direction of tubules is not completely straight which is caused by the presence of two tooth roots.

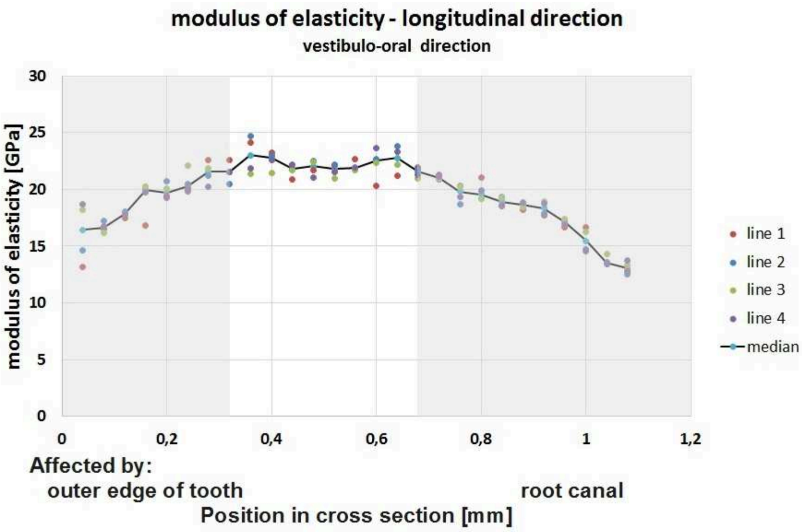

Fig. 3 Evolution of elastic modulus measured in longitudinal direction on transversal tooth cut (A).

Measurement of the modulus of elasticity on the longitudinal cut (in apical direction from the tooth neck towards the end of the tooth root, C) was divided into several submatrices of indents which were subsequently merged into one long matrix. The highest values of ~24 GPa were measured at the tooth neck-tooth root interface which is probably caused by changing in orientation of tubules in this part and dentin anisotropy. In the area along the root, the development of the modulus of elasticity was rather constant and the values ranged between ~20 GPa and ~15 GPa (Fig. 4).

We expect that the qualitative differences between the mean moduli in inner parts of the two sections i.e. 21,3 GPa (transversal cuts A, B) and 17,8 GPa (longitudinal cut, C) are caused by an intrinsic anisotropy of the intertubular dentin. The values of elastic moduli are, in this case, only indicative since theory of an anisotropic indentation [5] would be appropriate to receive quantitative results of the elastic constants.

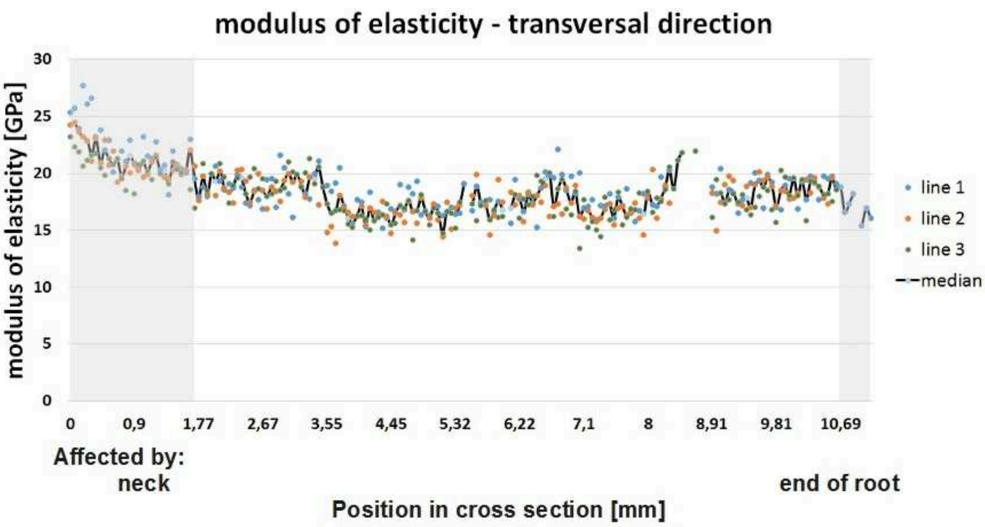

Fig. 4 Evolution of elastic modulus measured in transversal direction on longitudinal tooth cut (C).

Conclusions

In general, the elastic characteristics of dentin performed in this study are comparable with those found in the literature [6, 7, 8]. Measurement of local profiles in two typical cross sections showed that both anisotropy and density dependence exists in the tooth. These dependences were preliminary quantified by using Oliver and Pharr methodology for isotropic materials. More detailed study and correlations of mechanical properties with microstructural tooth features are planned as future developments.

Acknowledgement

The paper has been supported by the Grants of the Czech Science Foundation (P105/12/0824) and Technology Agency of the Czech Republic (TA03010886).

References

[1] Phillips R. W., Dental materials, eleventh ed., Science of dental materials 2011.

[2] Angker L., Swain M.V, Nanoindentation: application to dental hard tissue investigations, J. Mater. Res., (2006) 1893–1905.

[3] Oliver W. C., Pharr G. M., An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, J. Mat. 7 (1992) 1564-1583.

[4] Oyen M. L., Nanoindentation hardness of mineralized tissues, J. Biomech., 38 (2006) 2699-2702.

[5] Reisinger A. G., Pahr D. H., Zysset P. K., Principal stiffness orientation and degree of anisotropy of human osteons based on nanoindentation in three distinct planes, J. Mech. Beh. Bio. Mat., 4 (2011) 2113-2127.

[6] Mahoney E, Holt A, Swain M, Kilpatrick N., The hardness and modulus of elasticity of primary molar teeth: an ultra-micro-indentation study. J. Dent. 28 (2000) 589-594.

[7] Oyen ML, Handbook of nanoindentation with biological applications, Pan Stanford Publ. 2011.

[8] Balooch G et al., Evaluation of a new modulus mapping technique to investigate microstructural features of human teeth, J. Biomech., 37 (2004) 1223-1232.