The influence of peritubular and intertubular dentin for the development of the micromechanical properties

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Abstract: This paper is devoted to micromechanical properties of a tooth in dentin part. Mechanical properties of dry dentin from the human tooth were measured by a method of nanoindentation. The indentation was performed by loading to the maximum force of 3 mN. The trend of properties was observed depending on distance from the nearest tubule, its location in a peritubular or intertubular dentin area. A huge number of indents were made during the experiment and mechanical properties of these indents were compared, especially reduced modulus and hardness.

Keywords: human tooth; dentin; nanoindentation; hardness; reduced modulus.

1 Introduction

The knowledge of microstructure and mechanical properties of biological material such as human dentin is important for understanding processes which are coming during life and for designing elements (dental implants or tooth filling).

Dentin which forms most of the crown and tooth roots consists of main matter from odontoblasts and dentin tubules with Tomes´ fibers. Diameter of tubules is 1-2,5 μm and its density is 20.000-50.000 tubules/mm2 [1]. Peritubular dentin (PTD) forms a wall around tubules (width ~ 1,0-2,5 μm) and is highly mineralized [2]. Instead of intertubular dentin (ITD) which forms material between tubules and is less mineralized with more collagen.

The importance of knowing mechanical properties of dentin led to many researches in the past. These were carried out by quasi-static and dynamic methods whose usage was limited. Regarding it, indentation started to be used. The indentation have been used to analyse hardness and Young´s modulus of human dentin since the last century [4,5]. New methods for obtaining more detailed results are tested now [6].

The research by the method of the nanoindentation enabled people to obtain micromechanical properties of segments of human dentin – ITD and PTD. Average values of the hardness and the Young´s modulus in PTD measured by different researchers have been almost the same. For example average value of the Young´s modulus ~ 29,8 GPa and 29,3 GPa [4, 7]. Much more differences have been recorded for ITD properties. There the position of indents is really important because values near enamel and a pulp can differ to 20% [4]. Similar results and differences were obtained by carrying out indents large enough to reduce influence of tubules sufficiently [8].

We present our research which deals with the trend of properties in PTD and ITD depending on the distance of the nearest tubule. Other authors mentioned decrease of properties from interface of tubules in PTD and constant value in ITD [7]. The results of our research just as [7] were conducted by the indentor with operating mode CSM and the Berkovich´s tip with the methodology of Oliver & Pharr [9].

2 Materials and Methods

2.1 Preparation of Specimens

Four specimens were prepared from four molars in total. Two of them were assigned for the measurement of human dentin properties. The cleaned dry molars were embedded in epoxy resin (Fig. 1) and cut into slices ~ 10 mm after induration. Subsequently, these samples were grinded and polished by abrasive papers and canvas (Fig. 2). We used ultrasonic cleaning in alcohol bath for disposal of fine impurity.

Plane section was transversal (indentation in longitudinal direction) which crosscut tooth roots. The position of cuts had been chosen with regard to visible defects which could cause changes in mechanical properties by reason of demineralization.

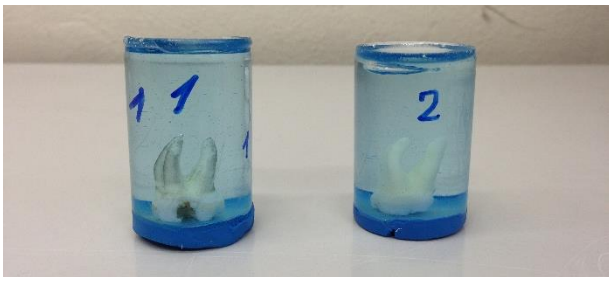

Fig. 1: The molars embedded in epoxy resin.

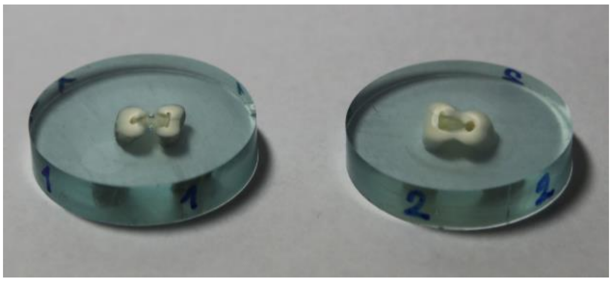

Fig. 2: Grinded and polished samples.

2.2 Nanoindentation

The indentation was made by the indentor CSM Instrument Nano Hardness Tester with Berkovich´s tip.

The first sample was used for obtaining an appropriate maximum force during loading. Seven matrixes were performed, each one with 25 indents. The maximum force for each matrix was different – 3, 5, 10, 15, 20, 25 and 30 mN. The penetration depth varied ~ 332 – 1163 nm. Force controlled tests were used for each cycle. The parameters of cycle: loading 120 mN/min, pause 10,0 s, unloading 120 mN/min.

Second sample which made for observing the trend of mechanical properties depending on the distance from tubule interface was loaded by the force of 3 mN. The penetration depth varied ~ 255 – 483 nm. The cycle parameters of the force controlled tests: loading 18 mN/min, pause 10,0 s, unloading 18 mN/min. The distance between indents was 4 μm regarding mutual affection.

3 Results

3.1 Test of Appropriate Maximum Force

7x25 indents were made for obtaining an appropriate maximum force during loading. Each matrix was loaded by a different force. From photos it was evaluated which indents had been located in ITD and which had interfered completely or partly with tubules. The most obvious difference in values of properties was found out if the lowest force had been used.

The hardness of ITD obtained by loading to the maximum force of 3 mN was ~ 973 MPa (Fig. 3). It was more than 30% increase of hardness compared to indents which had interfered with tubules. The differences were less obvious during usage of higher forces. It was caused mainly due to the size of indents which had interfered in ITD, PTD and tubules.

Regarding these results, the maximum force of 3 mN was chosen for next test. That could provide quality results and the penetration depth was lower so it could enable more exact location of indents in dentin – tubules, PTD or ITD.

Fig. 3: Average values of the hardness obtained during loading different forces

	H_{IT} [MPa]						
Maximum force [mN]	3	5	10	15	20	25	30
ITD	973,17	1038,86	1033,90	1022,06	1122,22	1017,97	980,18
tubules	658,15	995,61	867,87	1078,59	1057,37	983,09	975,39
difference	315,02	43,25	166,06	56,53	64,85	34,88	4,79

Fig. 4: Average values of the reduced modulus obtained during loading different forces

	E_r [GPa]						
Maximum force [mN]	3	5	10	15	20	25	30
ITD	33,90	31,80	28,40	27,26	28,66	27,50	26,72
tubules	27,10	31,50	27,57	28,25	27,78	27,56	27,10
difference	6,80	0,30	0,83	0,99	0,88	0,06	0,38

3.2 Test of Properties Trend

The indentation for obtaining the trend of properties in PTD and ITD depending on the distance from the nearest tubule interface required a huge number of indents. Two matrixes were made (8x8 and 6x6), 100 indents in total. The matrixes were located near the pulp where density of tubules was higher. According to photos, the indents in ITD were put into six sets (Fig. 5). The indents which interfered completely or partly into tubules were marked as the set 0.

Fig. 5: Putting indents into sets according to distance from tubule interface.

Set	1	2	3	4	5	6
distance from tubule interface [μm]	0 - 1	1 - 2	2 - 3	3 - 4	4 - 5	5 - 6

After evaluation of the sets, it was obvious that values of observed properties were increasing with the distance. The observed properties were the hardness (H_{IT}) and reduced modulus (E_r). It was obvious that the values of observed properties were increasing with bigger distance. The observed properties were the hardness (H_{IT}) and the reduced modulus (E_r). The first set indicated the average hardness ~ 965 MPa as the last set ~ 1222 MPa (Fig. 6, 7). It is increase more than 20%. Likewise the reduced modulus of first set ~ 25,8 GPa, the last set ~ 29,2 GPa (Fig. 6, 8).

The high standard deviation of the set 0 (the indents which interfered into tubules) is probably caused by various volume of interference in these tubules. The high values of hardness were reached if the indents had been located mainly in PTD.

Fig. 6: Average values of the hardness (H_{IT}) and the reduced modulus (E_r) of each set.

Set	0	1	2	3	4	5	6
H_{IT} [MPa]	837,77	965,58	1100,10	1162,66	1205,97	1162,17	1222,57
E_r [GPa]	24,94	25,75	27,20	27,56	28,37	28,37	29,24

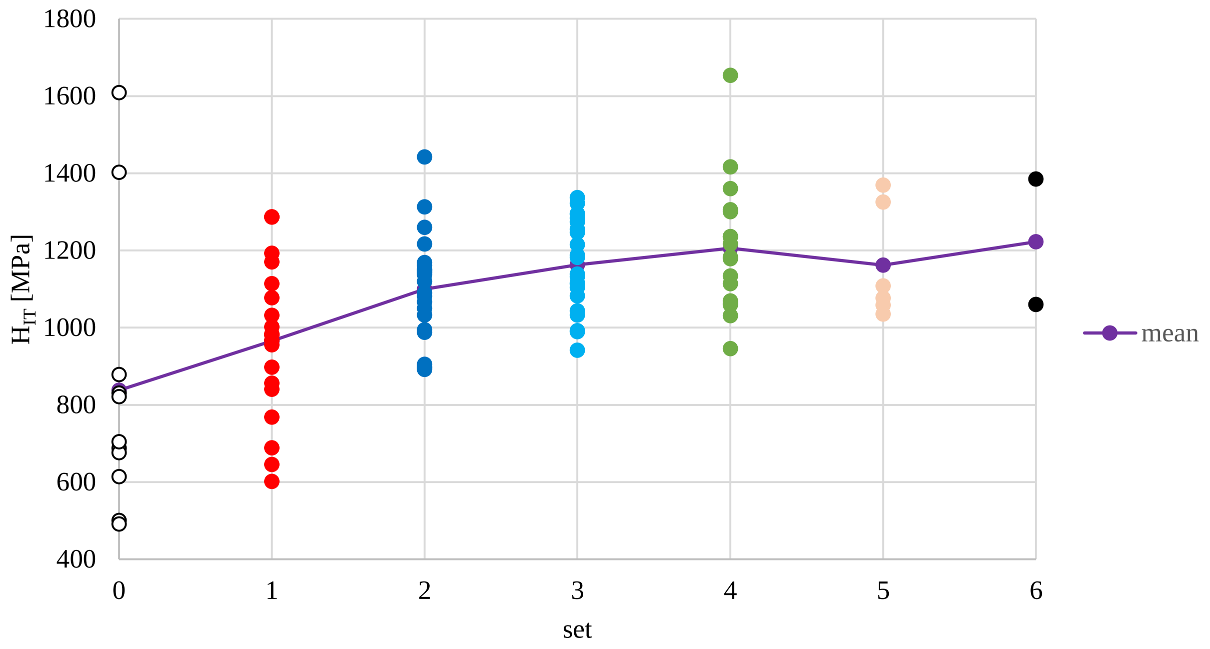

Fig. 7: The trend of the hardness in dentin part depending on the distance from the nearest tubule.

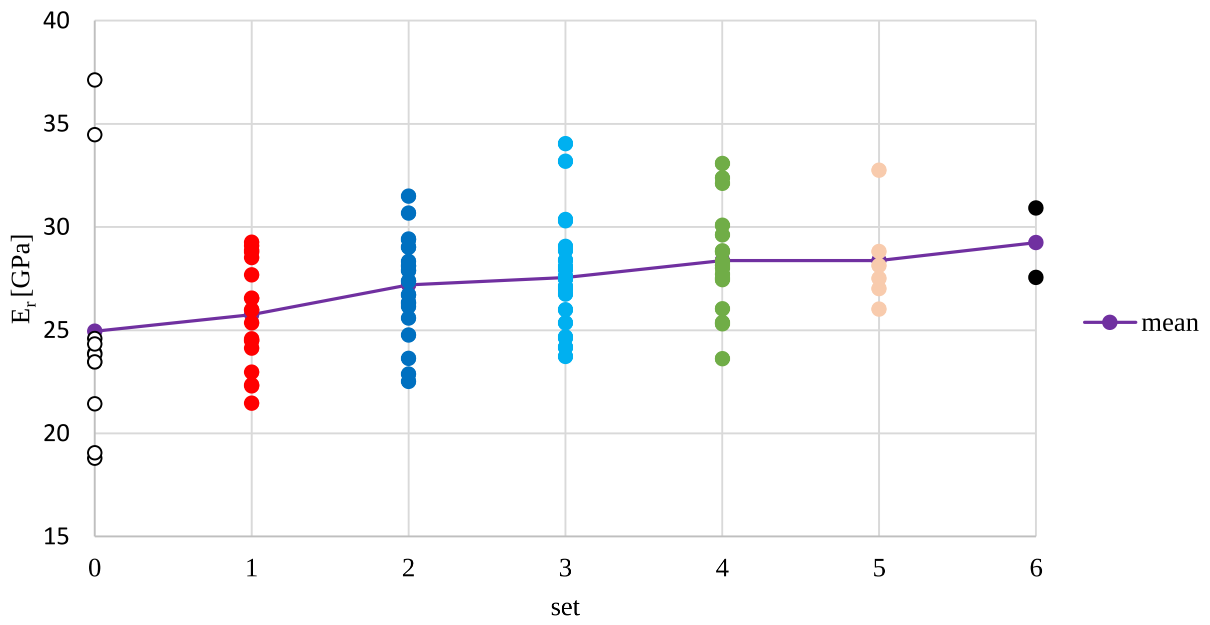

Fig. 8: The trend of the reduced modulus in dentin part depending on the distance from the nearest tubule.

The results show different trend than it has been performed by other authors so far. The authors point out decreasing trend of the observed properties in PTD and almost constant values in ITD [7]. The trend of the hardness and the reduced modulus obtained in this test can be assigned to ITD inhomogeneity and its affection by presence of tubules.

4 Conclusion

The influence of the distance from tubule interface for the development of the micromechanical properties in ITD was published in this paper. Firstly, the appropriate force for loading had to be found out to obtain optimal results and small indents. The maximum force of 3 mN was chosen for the next test. Subsequently, 100 indents were made. The increasing trend of the hardness and the reduced modulus from tubule interface was obtained. It is the different trend than have been published by other authors so far. They have denoted almost constant values in ITD. Because the measurement had been located near the pulp where the tubule density had been higher, the values of the properties were obtained only to the distance of 6 μm from the nearest tubule interface. In the next test it would be good to verify if the trend continues with the same character even further.

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