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Determination of Fracture Toughness of Human Enamel by Nanoindentation Based on Energy Release Method

HÁJKOVÁ Petra[1,a], JÍRA Aleš[1,b]

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

[a]petra.hajkova@fsv.cvut.cz, [b]jira@fsv.cvut.cz

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Abstract. Enamel is a brittle material. The main property showing its fracture resistance is fracture toughness. The aim of this study is to determine parameters necessary to estimate fracture toughness by the nanoindentation method. The method of fracture toughness evaluation based on dissipation of energy is described. In the first section of study, changes in values of hardness (H_{IT}) and reduced modulus (E_r) are observed with regard to crack initiation and growth. The downward trend of H_{IT} was found with the increasing peak load. The next section is focused on the loading rate impact on crack formation. The results supported the assumption that higher rates of loading induce greater crack formation.

Introduction

Enamel, like dentin or pulp, is an integral part of human teeth. Each of these parts has a specific function with corresponding material properties. The enamel protects the internal part of the tooth and is strained the most during mastication. This is the reason why it has the highest hardness among human tissues. Unfortunately, the high hardness is usually related to brittleness. The main property showing fracture resistance is fracture toughness.

The problem of enamel fracture toughness determination is that small volume of specimens limits the application of conventional methods. Consequently, the indentation method started to be used. In addition to fracture toughness determination, the method enables evaluation of other properties of enamel such as hardness or Young´s modulus.

The most frequently used method of fracture toughness determination based on indentation is Vickers indentation fracture (VIF) test. VIF enables direct determination of static fracture toughness (K_{IC}) from applied load and crack length originated in indentation. VIF was first introduced by Palmqvist in the 1950s [1], but it was advocated in 1976 by Evans and Charles [2]. Although it was supposed to evaluate fracture parameters of hard metals and ceramics, the method spread fast to the field of biomaterials. The main reason is the possibility of usage of small specimens and a survey of material properties at the micro level. However, in the last years the method has been criticised due to unreliable results. Authors [3, 4] have pointed out a large number of K_{IC} equations which rarely provide the same results. Even so, most papers [5, 6] evaluate fracture toughness of enamel by VIF. Rarely, authors [7] apply other methods as single edge notched bending (SENB) specimen. The unreliability of VIF led to research of other methods for fracture toughness determination. Kruzic et al. [8] compare application of different methods (VIF, CCIF, VCOD and IIF) aimed at fracture toughness measurement of biomaterials. Hrbek, Malzbender et al. [9-11] deal with the possibility of determining fracture properties and erosion behaviour by a method based on dissipation of energy during indentation.

The aim of this study is to determine parameters necessary to estimate fracture toughness of enamel by the method of nanoindentation. The nanoindentation method based on dissipation of energy would reduce some undesirable aspects which are found to be wrong in VIF application. Additional advantage is the use of nanoindentation instead of microindentation. It allows to load the specimen by smaller maximum forces, so as to examine each part and trend in the enamel more precisely. In this paper, changes in values of hardness (H_{IT}) and reduced modulus (E_r) are observed with regard to crack initiation and growth. The next goal is an assessment of loading/unloading rate impact on extent of damage during indentation with the same peak load.

Materials and Methods

Preparation of Specimens. A sample was prepared from a molar which was extracted due to orthodontic reasons. The clean, dry molar was embedded in Dentacryl. A 15 mm thick section was sliced perpendicularly to the longitudinal axis of the tooth by a water-cooled saw (ATM, Germany) equipped with a diamond blade. A required depth in enamel was reached by grinding by abrasive paper (grit size of 320). Then, the sample was polished by finer abrasive papers (grit size of 1000, 2500) and a diamond paste (0,25 μm).

Nanoindentation. The indentation was performed on the indenter CSM Instruments Nano Hardness Tester (Anton Paar, Australia) with a wedge tip. The methodology used for determination of hardness and reduced modulus was described by Oliver and Pharr [12]. Five indentation matrices were performed for observing changes in values of H_{IT} and E_r with regard to crack initiation and growth. Each matrix consisted of 16 indents. Force controlled tests with a different peak load were carried out for each cycle. The maximum forces were 10, 20, 40, 80 and 150 mN. The parameters of each cycle: loading 120 mN/min, pause 10.0 s, unloading 120 mN/min.

The next four matrices were performed for the comparison of loading/unloading impact on extent of damage. Three matrices were located in enamel, the last one in dentin. Each matrix consisted of 25 indents. Force controlled tests with a maximum force of 150 mN were carried out for each cycle. The loading and unloading rate was different for each matrix – 60, 250, 450 mN/min for enamel and 450 mN/min for dentin.

The axial distance of the indents was selected with regard to the contact area size of the indents. The position of indents towards the dentino-enamel junction (DEJ) and occlusal surface differs a lot due to different sizes of the matrices.

Energy Release Method. Energy release method is an alternative method for fracture toughness evaluation. It enables calculation of K_{IC} using the energy dissipated during indentation. The total work of indentation (W_{tot}) can be easily obtained by integration of the loading curve. Consequently, the total work of indentation can be divided into reversible (W_{rev}) and irreversible work (W_{irr}) (Eq. 1) or more different energy dissipation mechanisms (Eq. 2) including work of fracture ($W_{fracture}$).

$$W_{tot} = W_{rev} + W_{irr} \tag{1}$$

$$W_{tot} = W_{plastic} + W_{elastic} + W_{fracture} + W_{creep} + W_{other} \tag{2}$$

Reversible and irreversible work can be as like as total work obtained from the load-displacement curve. Fig. 1 shows that W_{rev} is the area under unloading curve, whereas W_{irr} is the area enclosed between the loading and unloading curve.

The load-displacement curve as shown in Fig. 1 can also indicate crack formation. In this case, errors (pop-in) in the loading curve appear, displacement grows suddenly (δ) and the loading curve is shifted to the right.

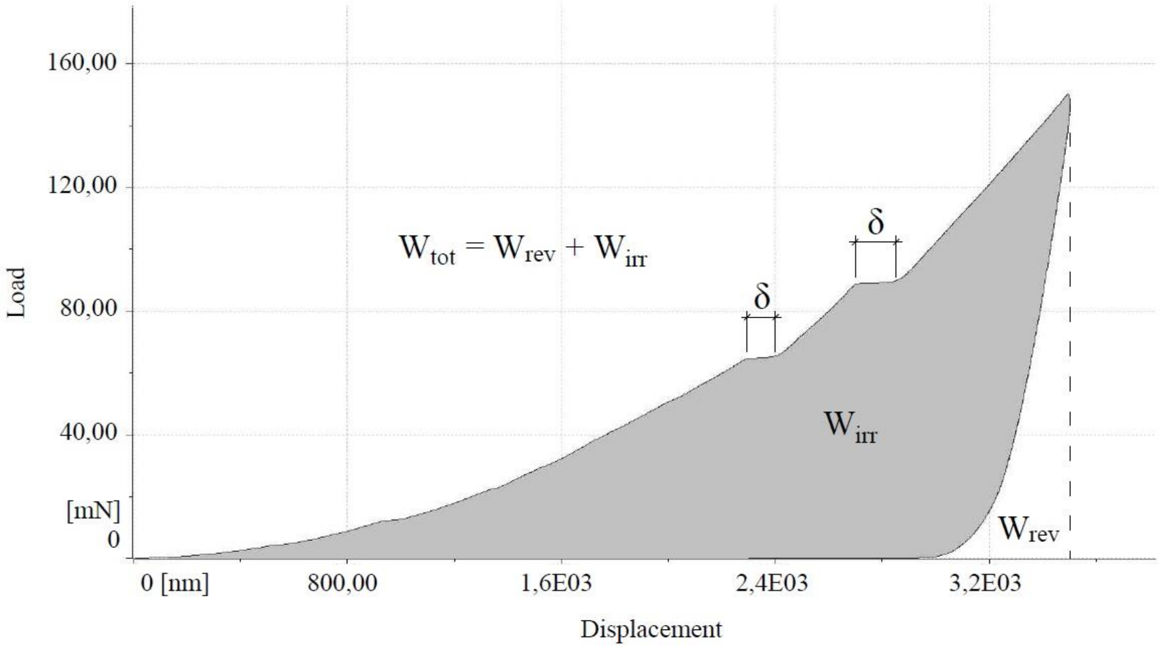

Fig. 1: Load-displacement curve showing total work (W_{tot}) divided into reversible (W_{rev}) and irreversible (W_{irr}) work. Shift of loading curve (δ) is caused by crack formation.

Results

Property Changes Related to Cracks. Changes of hardness (H_{IT}) and reduced modulus (E_r) were observed with regard to crack formation. Five indentation matrices with 16 indents were performed by force controlled test with a different peak load for each matrix. Table 1 shows mean values of H_{IT} and E_r. There were significant changes of properties. It was observed that the hardness was the greatest for the minimum loading force of 10 mN ~ 3791 MPa and decreased up to ~ 3246 MPa for the maximum loading force of 150 mN. Reduced modulus showed the opposite trend. Fig. 2 shows variation of H_{IT} and E_r with the indentation peak load.

Table 1: Mean values of observed properties (H_{IT}, E_r) and ratio of irreversible (W_{irr}) to total (W_{tot}) obtained by force controlled tests with the different peak load.

Maximum force [mN]	10	20	40	80	150
H_{IT} [Mpa]	3790.40	3707.57	3498.63	3275.21	3246.29
E_r [Gpa]	79.65	80.33	80.95	81.55	85.66
W_{irr}/W_{tot} [-]	0.874	0.880	0.884	0.887	0.889

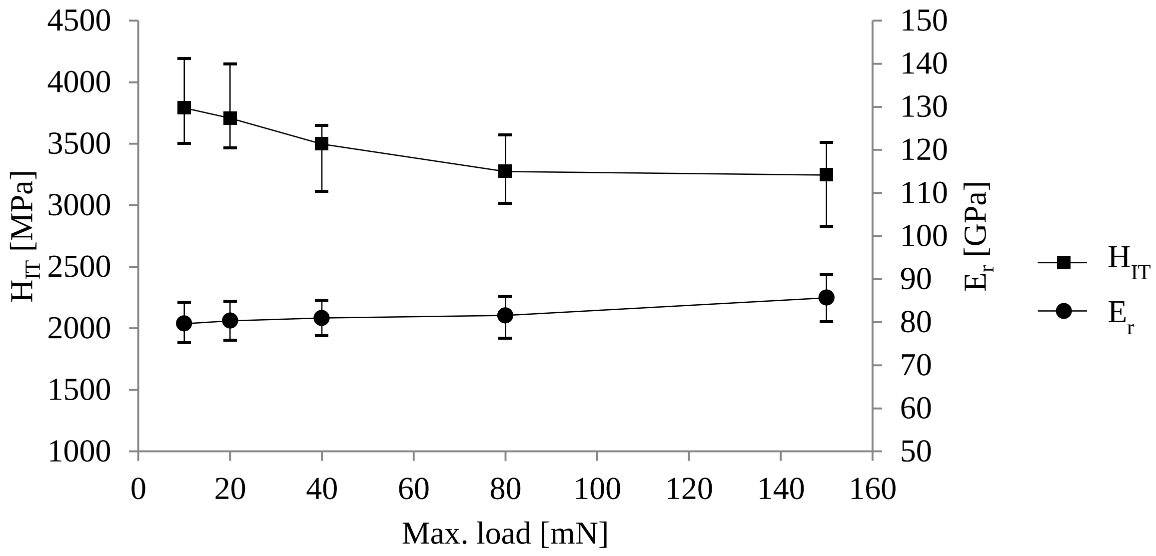

Fig. 2: Variation of hardness (H_{IT}) and reduced modulus (E_r) connected to the maximum loading force.

Compared with our last study [13], enamel showed significant changes of H_{IT} whereas hardness of dentin seems to be almost constant with the increasing maximum loading. Property changes in enamel respond to pop-in events observed in load-displacement curves and increasing ratio of W_{irr} to W_{tot} which indicates crack initiation and growth. Dentin which is much less brittle material does not show these characters.

Loading Rate Impact on Crack Formation. Three indentation matrices with 25 indents were performed to assess the loading/unloading rate impact on crack formation. The fourth matrix was located in dentin to obtain property values for illustrative comparison of dentin and enamel. Just as in the first parts of study, hardness (H_{IT}) and reduced modulus (E_r) were observed. In addition, Table 2 shows the mean reversible (W_{rev}), irreversible (W_{irr}) and total (W_{tot}) work of indentation and ratio of W_{irr} to W_{tot}. The changes of H_{IT} and E_r were not as significant as in the test of property changes which showed a difference between formed cracks as a result of greatly varying peak loads. But there was a decline in H_{IT} from ~ 3287 MPa at the loading rate of 60 mN/min to ~ 3202 MPa at the loading rate of 450 mN/min. In contrast to the first section of the study, values of E_r decreased.

Table 2: Mean values of observed properties (H_{IT}, E_r), work of indentation and ratio of irreversible (W_{irr}) to total (W_{tot}) work obtained by force controlled tests with different loading/unloading rate.

Loading rate [mN/min]	Enamel			Dentin
	60	250	450	450
H_{IT} [Mpa]	3286.60	3309.95	3201.79	1153.21
E_r [Gpa]	85.33	86.16	82.22	24.99
W_{rev} [pJ]	18613	16741	15634	27773
W_{irr} [pJ]	167390	168153	181320	310837
W_{tot} [pJ]	186003	184894	196955	338610
W_{irr}/W_{tot} [-]	0.899	0.909	0.920	0.918

The upward trend of the W_{irr}/W_{tot} ratio connected with increasing loading/unloading rate supports an assumption that higher rates induce greater crack formation. The extent of damage can be observed in Fig. 3 as well.

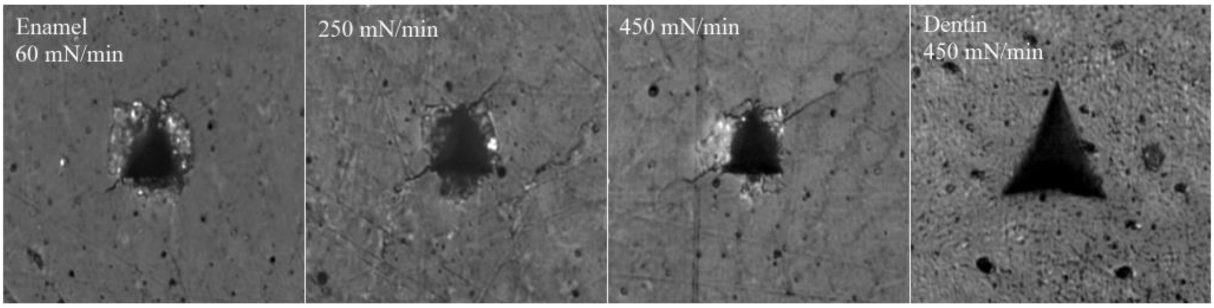

Fig. 3: Indents performed by force controlled test with the maximum force of 150 mN. Loading/unloading rate of indentation - 60, 250 and 450 mN/min. There are observable cracks in enamel. In contrast, the fourth indent was located in dentin where no cracks were recognized.

Conclusions

The parameters for fracture toughness determination were obtained. Although the energy release method is based on constant material properties, it was suggested that fracture events affect hardness (H_{IT}) and reduced modulus (E_r) in the first section of the study. It was compared to the study of dentin which did not show any significant changes. The upward trend of the W_{irr}/W_{tot} ratio connected with the increasing peak load indicated the crack initiation and growth. It was supported by load-displacement curve errors (pop-in) and investigation of indents under a microscope. In the next study, it is necessary to focus more on the relationship between H_{IT}/E_r and W_{irr}/W_{tot} which appear often in fracture toughness equations.

The loading rate impact on crack formation was observed in the second section of the study. The results showed a greater W_{irr}/W_{tot} ratio when the enamel was loaded at higher loading rates. It supported the assumption that higher rates induce greater crack formation. There was a decrease of H_{IT} ~ 2.6% and a decrease of E_r ~ 3.7% by performing indents at the highest rate of 450 mN/min and the lowest rate of 60 mN/min.

This study precedes the fracture toughness evaluation. The problem of work of fracture ($W_{fracture}$) extraction will be crucial. It is also necessary to suppress other impacts on crack formation - creep, test conditions etc. as much as possible. The next issue will be the identification of fracture geometry using computed tomography scanning (CT scan).

Acknowledgement

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