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Influence of dental materials on hardness and Young's modulus of the surface layers of tooth enamel formed as a result of friction

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Purpose: The purpose of this work was to determine the influence of dental materials used as permanent fillings on the mechanical properties of the tooth enamel surface layer subjected to friction with these materials. *Methods:* Dental composite materials (five types) were differentiated in terms of size and shape of the filler particles and matrix type over the course of tests on the chewing simulator under two different loads set during friction. Next, it was measured values of wear and nanoindentation for the resulting friction rates on the enamel (3 different load ranges). *Results:* It was found that the enamel's resistance to tribological wear is significantly higher than that of the tested dental materials. It is also important to note that, depending on the penetration depth of the indenter (depends on the indenter pressure), different hardness values and Young's modulus of enamel were obtained after friction with different dental materials. This demonstrates the formation of a surface layer with different properties than the native material. *Conclusions:* Analysis of the obtained results suggests the existence of different tribological wear mechanisms, as evidenced by significant differences in the wear values of dental materials and enamel. The data show that the enamel surface layer modified by the contacting dental material is shaped to a certain depth, and different thickness ranges of the changed layer have different properties.

Key words: *enamel, hardness, dental material, wear, nanoindentation, Young's modulus*

1. Introduction

The literature presents a well-documented empirical opinion [9] that, under friction-related conditions far from the thermodynamic equilibrium of friction processes, the surface layers of contacting bodies are modified and entirely new structures, so-called secondary structures, are created. The properties of these structures differ completely from the native material. Their presence on the surface of enamel and dental materials has been reported in previous papers [15], [17], [20]. It seems that studying the mechanical properties of secondary structures formed on enamel has significant potential for explaining the tribological behavior of enamel in contact with dental materials.

Changes in durability parameters are observed depending on the contact depth of the layer, and the largest differences are visible closest to the surface [17].

Understanding the mechanical properties of the thin secondary layers formed during friction processes (tribological pair: enamel–dental composite) can give promising results, suggesting a practical direction for modification of the composition of available composites. Based on this assumption, the authors attempted to determine the hardness and Young's modulus of human enamel as a response to the conditions and work environment. The mechanical properties and depth range of changes in the surface layers of the enamel were determined using the nanoindentation technique.

Dental enamel is the hardest and most mineralized tissue in our body. Therefore, it also has a number of

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specific properties. Specific properties of enamel are revealed during chewing and biting, when the enamel carries heavy loads [4], [5], [21] and exhibits high wear resistance during prolonged chewing and biting processes that occur in the oral cavity over the course of a human lifetime.

Due to the high variability of the features of components in dental composites (type of matrix and type, size and shape of the filler), which may affect different characteristics of tribological processes and result in varying wear volumes. It is important to analyze the tribological interactions of different dental materials with the enamel surface. According to study [13], the tribological properties of composite materials depend, to a large extent, on their chemical composition. The results of the study and analysis of its results allows to evaluate the tribological wear resistance of selected dental materials and to determine the factors that intensify the wear of hard dental tissues during contact with these materials. Under natural conditions, in the process of biting and chewing, the tooth contacts the opposite tooth in the stomatognathic system. Abrasion of co-operating surfaces occurs. The average wear on contacting teeth occurring during normal biting and chewing processes tested *in vivo* is approximately 29 μm for the molars and 15 μm for the premolars over the course of a year [10]. In the cases where defects in the hard tissues of the opposing tooth are filled with dental material, the enamel wear is more intense than during enamel-on-enamel friction. A study by Söderholm and co-workers [18] testifies to this effect. Enamel wear values are within the range of 10–40 μm/year, while the wear of dental materials is 8–9 μm/month (96 –108 μm/year) [11]. Wear values for dental materials are nine times higher than for enamel. This shows that there is an existing problem in terms of the durability of filling materials. Such rapid wear causes damage to the dental filling, which may even require replacement. However, this requires cleaning of the cavity and removal of more dental tissue. This problem is important to all of us, because after the collapse of the permanent teeth, no further toothing will grow to replace them. The healthier the teeth, the more comfortable our lives. The clinical significance of the research undertaken by the authors is therefore highlighted, especially since the research on changes of enamel's mechanical properties in the context of its wear resistance is relatively low, and there are still issues to be clarified.

Table 1. Characteristics of light-curing dental materials used in the experiment

No.	Material/ manufacturer	Filler			Resin type	Description of the material
		Type	Volume content [%]	Particle size		
1	*BOSTON/ DENTOMAX/ Arkona*	glass Ba-Al-Si, fire silica, TiO_2	69	0.72 μm (mean)	Bis-EMA, UDMA, EGDMA	Composite with increased mechanical strength
2	*SDI ICE/Sdi*	inorganic	60	0.04–1.5 μm	multifuncional methacrylic ester	Universal nanofilter composite: (hybrid technology + nanotechnology)
3	*ARTISTE/ DENTOMAX Pentron Clinical*	nanocomposite Si-Al-Ba	66	0.02–0.7 μm	not specified by producer	nanohybrid composite
4	*SUPER COR/ DENTOMAX Spofadental*	microhybrid, inorganic	59	0.6 μm (mean)	not specified by producer	microhybrid composite
5	*FILTEK TM SUPREME/ 3M*	Molecules SiO_2 ZrO_2, complexes SiO_2/ZrO_2	55.6	5–75 nm and claster 0.6–10 μm	Bis GMA, UDMA, TEGDMA, PEGDMA, bos-EMA	nanocomposite

Table 2. Explanation of the abbreviations of resins included in the dental materials used in the experiment

Resin type	Bis-GMA	UDMA	TEGDMA	EGDMA	PEGDMA	Bis-EMA
Full name	glycidyl phenyl methacrylate	dimethacrylate resin: diurethane dimethacrylate	dimethylacrylate resin: triethylene glycol dimethacrylate	ethylene glycol dimethacrylate	ethylene pentaglycol methacrylene	bisphenol A diglicydoeter dimethacrylate

2. Materials and methods

2.1. Materials

In the experiment that was conducted, several composite materials used as permanent dental fillings were tested. The physicochemical characteristics of the dental materials used in the experiment are shown in Table 1. The filler type, particle percentage, particle size and matrix type were determined. Abbreviations corresponding to matrix types are explained in Table 2.

Suitable prepared samples of composite materials in the shape of a truncated cone were used for tribological studies. The enamel was obtained from molar and premolar teeth, removed for orthodontic reasons. The teeth were cut using a diamond water-cooled circular saw. Teeth prepared in this manner were then stiffened in resin and sanded using 800 to 2000 granulation sandpaper. The samples were then polished using aluminium oxide. Enamel samples were kept in Hanks' solution at a temperature of about 5 °C until the time of the test to prevent excessive dehydration and to keep their mechanical properties at an adequate level [8].

The light-cured composites used for permanent dental fillings were polymerized according to the manufacturer's instructions using Blue Cap 1000 LED Curing Light (Dentazon), then glued to the aluminum holder and grinded into cones with an angle of 30°. The next step was to grind cones (grade 2000 sandpaper) and polish them with a cloth to obtain a mirror surface. Sample diameters were measured prior to the experiment.

2.2. Methods of investigations

2.2.1. Character of investigations

It is advisable to carry out *in vitro* studies of enamel and other elements existing in the oral cavity in the environment of saliva, because simulating the natural working conditions of the stomatognathic system is an important element of these studies. Artificial saliva is used in the study of wear in friction pairs like enamel-dental material as a lubricant and as a moisturizer for enamel [1], [12], [14]. Saliva performs many functions in the human body. One of its more important functions is lubrication, which decreases the friction between elements of the human stomatognathic system [2]. In this work, tribological tests were performed in an artificial saliva environment prepared according to the Fusayama formula with Holland's modification [6]. The composition of artificial saliva is shown in Table 3.

The experiment was carried out on a chewing process simulator, which performs cyclic semi-sinusoidal loading of the countersamples, which were made of dental material used for dental permanent fillings and embedded in an aluminum holder, with reciprocal movement of a sample made of half of a human tooth embedded in resin (Fig. 1). The device was designed and made at the Faculty of Mechanics of Białystok University of Technology. A detailed description of the device is available in another author's work [16].

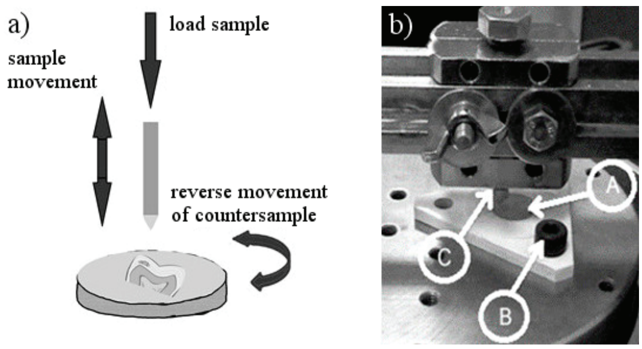


Fig. 1. (a) Sample and countersample movements, (b) photograph of friction testing unit: A – countersample (composite tested), B – fixing screw, C – sample

The load for the tribological experiment was chosen to represent the load acting on the tooth during dental work [4]. Two tribological experiments were performed for each composite with cyclic loading of 7.7 N and 23.5 N. The test conditions are described in Table 4.

Table 3. Composition of Fusayama artificial saliva with Holland modification [4]

Reagent for 1 l	KCl	NaCl	$CaCl_2 \cdot 2H_2O$	$NaH_2PO_4 \cdot 2H_2O$	$Na_2S \cdot 9H_2O$	Urea
Quantity [g]	0.4	0.4	0.795	0.78	0.005	1

Table 4. Description of test conditions

Composite	Quantity: 10; type: 5; shape: truncated cone
Dental enamel of human	Shape: half tooth
Load during friction test	Alternating semi-sinusoidal: load 7.7 N and 23.5 N
Test parameters	Movement direction of the sample: vertical, countersample movement: reciprocating, single testing time: 3 hours, frequency of sample movement: 1.58–1.62 Hz, sample separation 2 mm, wear track length: approx. 2.5 mm, environment: artificial saliva, temperature: ambient (20–23 °C)

2.2.2. Wear

The volume lost of test material was measured with available measuring devices. A confocal laser microscope was used for enamel wear measurement, which, compared to a profilometer, proves to be a more effective measuring device in terms of speed of measurement [19]. The sample is placed on the table of the microscope and scanned by a laser beam. Data is automatically saved into compatible software. In contrast, the wear of dental composite was calculated by substituting measured values into the formula (1) (truncated cone diameter, taper angle) before and after the test (Fig. 2).

$$Z_0 = \frac{tan\alpha\pi}{24}(D_1^3 - D_0^3)[\text{mm}^3] \quad (1)$$

where:

Z_0 – volumetric wear,

$\tan\alpha$ – tangent of the cone angle,

D_0 – the diameter of the sample tip before the wear test,

D_1 – the diameter of the sample tip after the wear test.

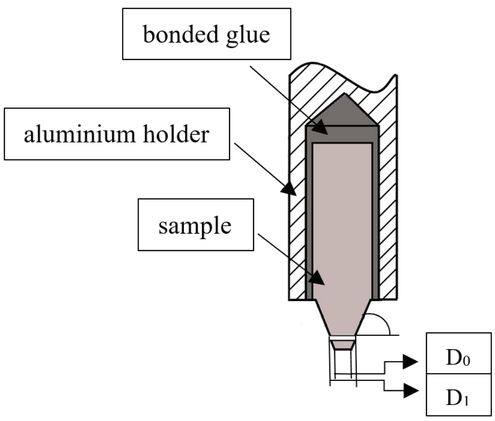


Fig. 2. Shape and dimensions of enamel samples (before testing)

2.2.3. Nanoindentation

After tribological wear tests the indentation experiments on the worn enamel areas and on unworn cross-sectional enamel were performed using a nano-based indentation system (CSM Instruments, Switzerland). A Berkowitch diamond indenter was used. Measurements were taken on the surface of the enamel within the friction trace area and comparatively on the non-friction surface at 1 mN, 10 mN and 40 mN indentation loads, resulting in hardness values and Young's modulus. Six indents were made for each indenter load.

3. Results

The collected data was statistically analyzed in Statistica.

3.1. Wear

Figure 3 shows the wear intensity values for the material used during the tribological test. Wear intensity is expressed here as the ratio of material wear during friction to the overall distance traveled by the sample. The load that was applied during friction was accounted for.

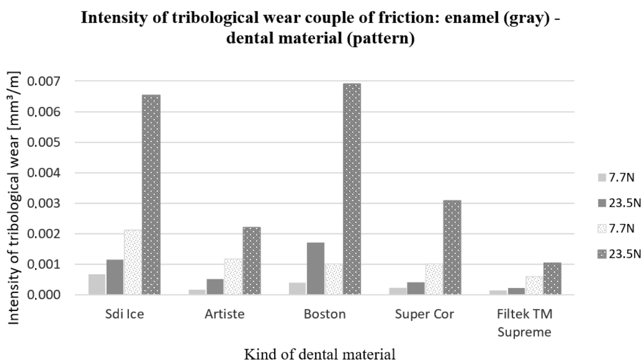


Fig. 3. Intensity of tribological wear couple of friction: enamel (gray) – dental material (pattern)

Differing intensity of wear (Fig. 3) can signify a differing nature of tribological processes for each material. This may be influenced by the structure of the dental material in contact with the enamel, since the conditions of the friction process remain constant for each tribological pair. Figure 3 shows significant differences in wear intensity between the enamel and dental material.

Table 5 distinguishes the maximum values obtained for each measurement group. There is a clear tendency for increased wear of enamel and composites at higher loads.

Table 5. Intensity of wear:
the value of enamel wear and dental materials

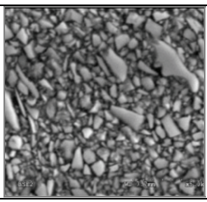
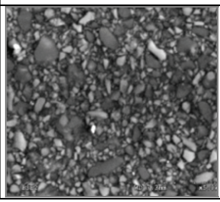
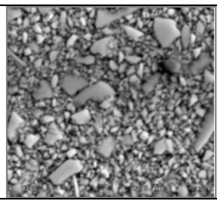
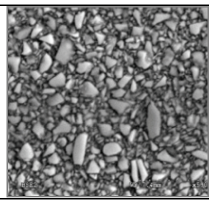
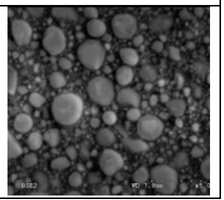
Intensity of wear [mm^3/m]				
Load during friction	7.7 N	23.5 N	7.7 N	23.5 N
Material	Enamel	Enamel	Composite	Composite
Sdi Ice	0.00066	0.00114	0.0021	0.0065
Artiste	0.00017	0.00051	0.0012	0.0022
Boston	0.00039	0.00170	0.0010	0.0069
Super Cor	0.00023	0.00040	0.0010	0.0031
Filtek TM Supreme	0.00014	0.00021	0.0006	0.0010

Magnified structures (5000 ×) of the dental materials obtained by scanning microscopy: SEM Hitachi S-3000 N are shown in Table 6.

3.2. Nanoindentation

A comparison of the hardness and elastic modulus obtained in various experimental modes (1, 10 and 40 mN) is shown in Figs. 4–8. Results of nanoindentation studies indicate that the hardness and Young's modulus of human enamel differ significantly between friction areas and areas not subjected

Table 6. Characteristics of dental materials and SEM images

Dental material	Sdi Ice	Artiste	Boston	Super Cor	Flitek TM Supreme
Particle size of the filler [μm]	0.04–1.5	0.02–0.7	0.72	0.6	4–20 and 0.6–10
Volume of filler [%]	60	66	69	59	55.6
Magnification 5000×					

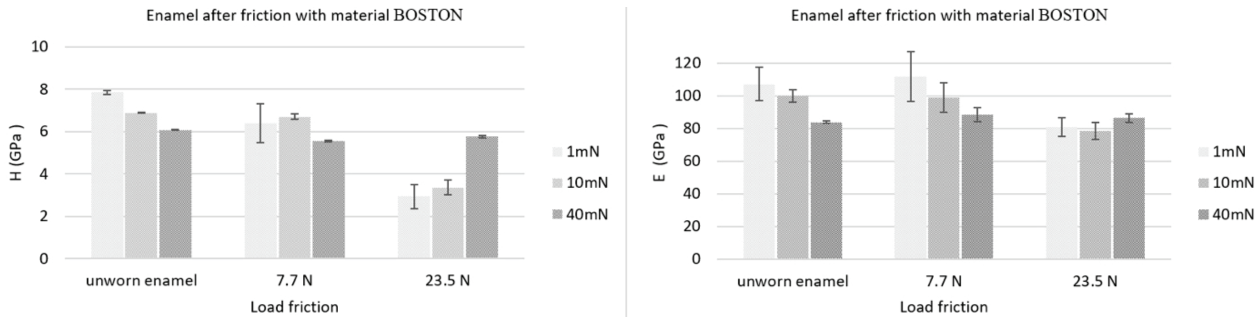


Fig. 4. Hardness (H) and Young's modulus (E) determined in the study (mean and standard error)
under indenter load 1 mN, 10 mN and 40 mN

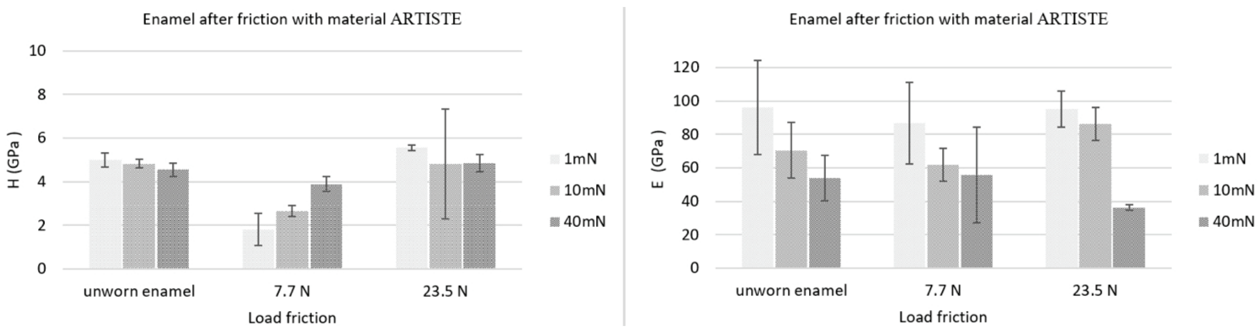


Fig. 5. Hardness (H) and Young's modulus (E) determined in the study (mean and standard error)
under indenter load 1 mN, 10 mN and 40 mN

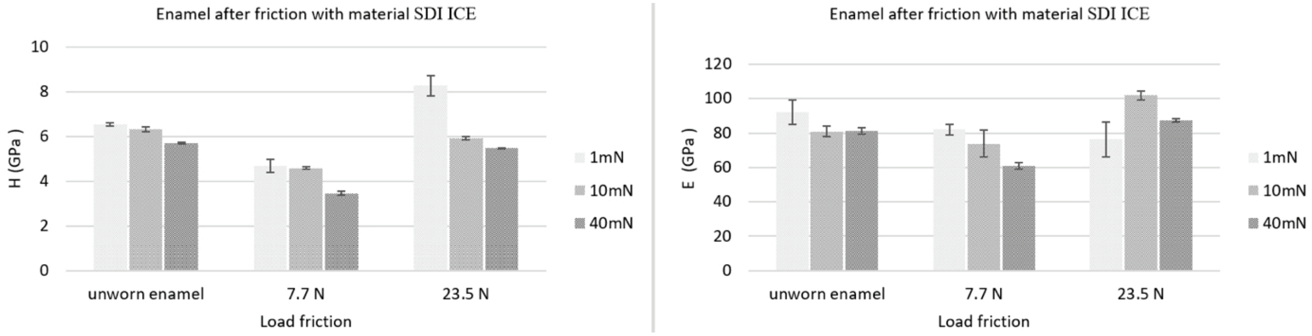


Fig. 6. Hardness (H) and Young's modulus (E) determined in the study (mean and standard error) under indenter load 1 mN, 10 mN and 40 mN

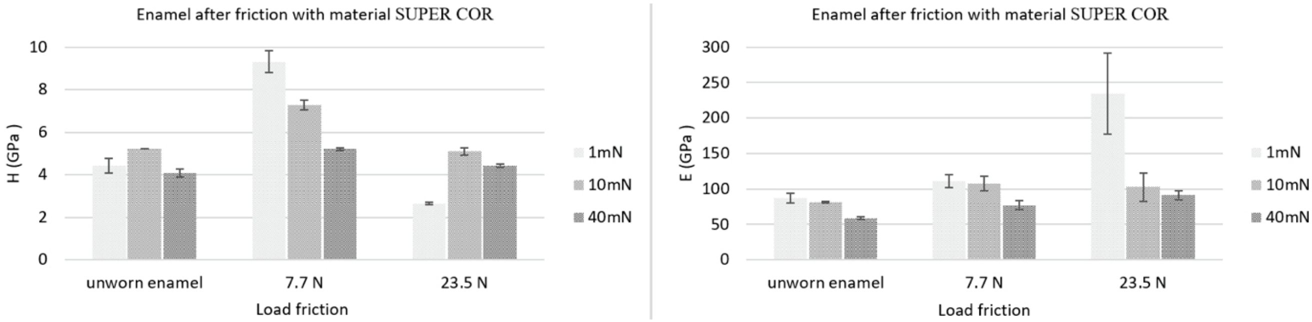


Fig. 7. Hardness (H) and Young's modulus (E) determined in the study (mean and standard error) under indenter load 1 mN, 10 mN and 40 mN

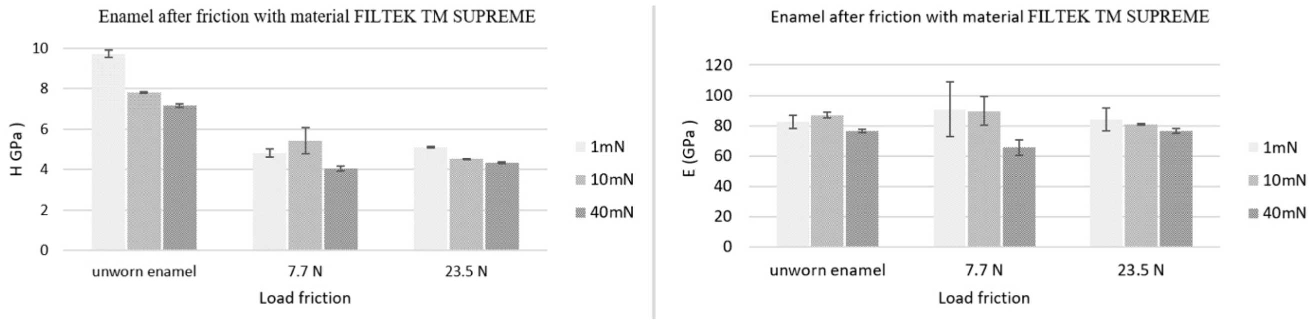


Fig. 8. Hardness (H) and Young's modulus (E) determined in the study (mean and standard error) under indenter load 1 mN, 10 mN and 40 mN

to tribological interactions. Hardness and Young's modulus values from friction areas under a load of 7.7 N are within the range of 1500–9000 MPa and 60–110 GPa, respectively, and under a load of 23.5 N, within the range of 2500–8100 MPa and 35–230 GPa, respectively.

The standard deviation for each sample was calculated to determine the measurement error in the distribution of the values obtained (H – hardness and E – Young's modulus) with respect to the mean using Statistica software. The characteristics of enamel's mechanical properties are shown in Figs. 9–13.

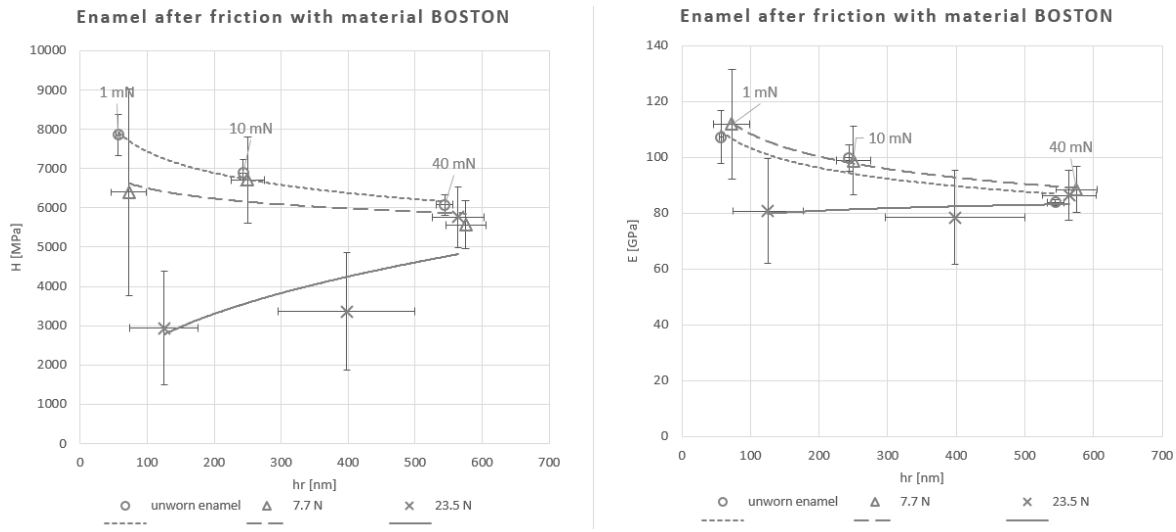


Fig. 9. Results of enamel nanoindentation with respect to penetration depth of indenter (mean and standard deviation)

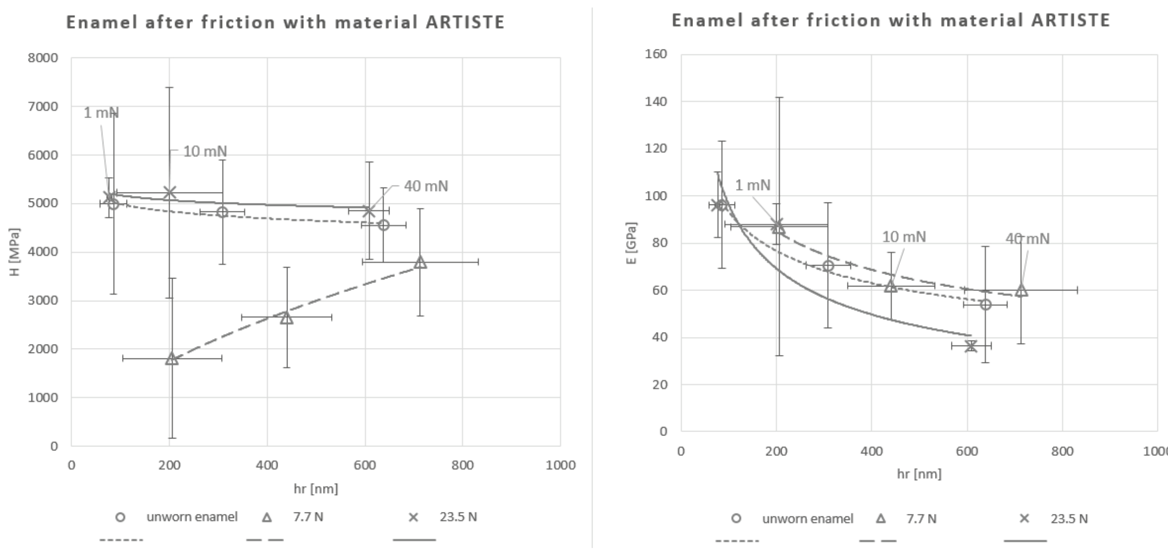


Fig. 10. Results of enamel nanoindentation with respect to penetration depth of indenter (mean and standard deviation)

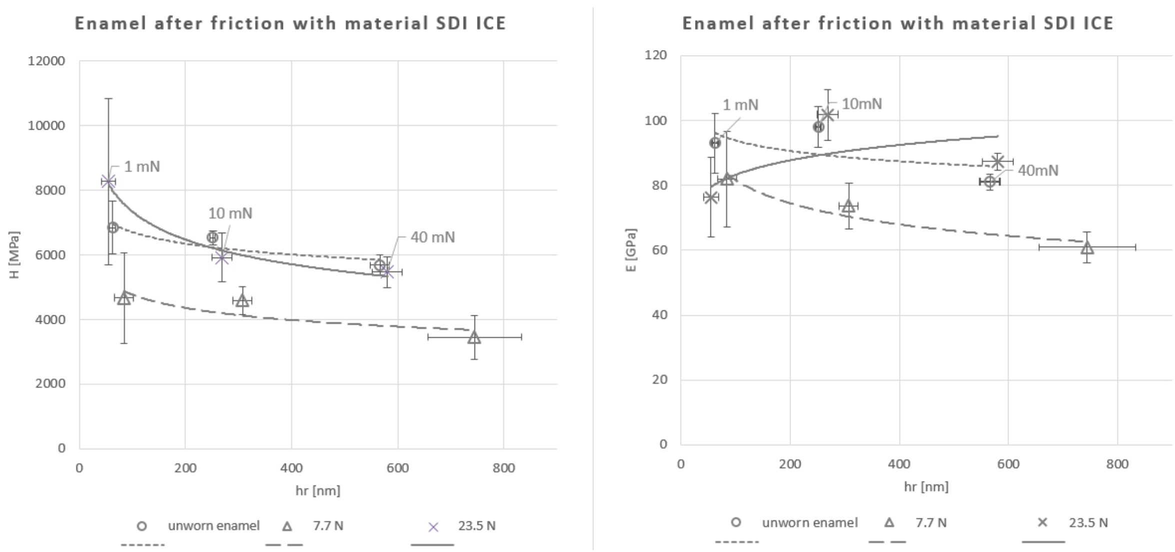


Fig. 11. Results of enamel nanoindentation with respect to penetration depth of indenter (mean and standard deviation)

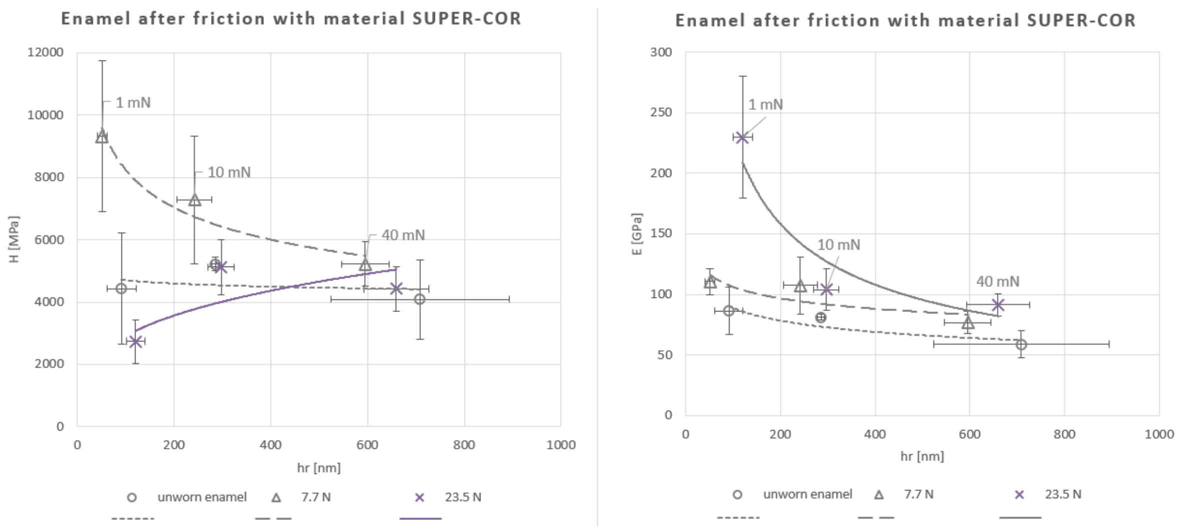


Fig. 12. Results of enamel nanoindentation with respect to penetration depth of indenter (mean and standard deviation)

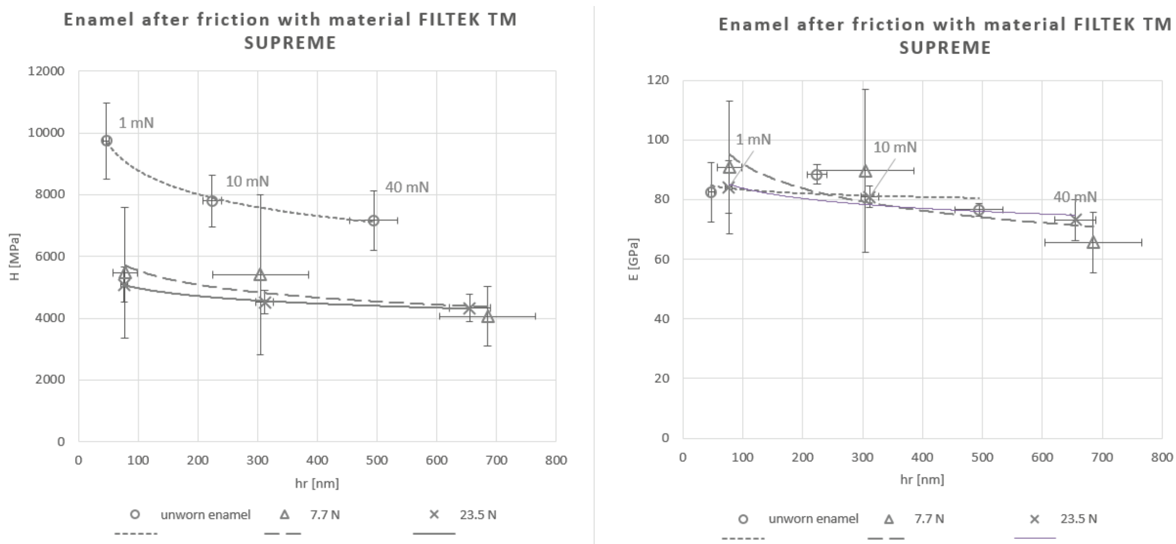


Fig. 13. Results of enamel nanoindentation with respect to penetration depth of indenter (mean and standard deviation)

4. Discussion

Differing intensity of wear (Fig. 3) can signify a differing nature of tribological processes for each material. This may be influenced by the structure of the dental material in contact with the enamel, since the conditions of the friction process remain constant for each tribological pair. The results of tests carried out by Elmaria et al. [22] indicate similar conclusions that the loss of enamel height varied significantly depending on the material contacted with it.

Table 5 presents the highest wear of enamel after friction at 7.7 N load in contact with Sdi Ice material and amounts to 0.00066 mm^3/m. In addition, as shown in the graph and Table 5, for this tribological pair, the dental material wears about 3 times more: the composite wear is 0.0021 mm^3/m. In this case, the different sizes of particle fillers (0.04–1.5 μm) can be significant, along with the largest particle size of the materials used and the shape of these particles, which appears to be the most oblong and sharp. It is assumed that abrasive wear by micro-grinding is dominant in this case.

It is interesting to note that the highest wear of enamel, with identical experimental conditions for all friction pairs, is in contact with the Boston material, in which the filler content is the highest, i.e., 69%, and the average filler particle size is 0.72 μm. This means that, in the case of the composites used, the proportion of resin is the lowest. Such a material structure can cause less elastic deformation upon loading, resulting in slight deformation of the particles within the resin volume, and thus, grinding becomes the dominant wear mechanism during friction in this case.

Much higher wear of the composite, compared to enamel wear, occurs for Sdi Ice and Boston materials at both load values, especially at a load of 23.5 N, and the difference is approximately 6- and 4-fold, respectively (composite wear of 0.0065 and 0.0069 mm^3/m). Considering the shape of the filler particles, it turns out that the spherical particles in the Filtek TM Supreme material cause the least enamel wear. The reason may be that, in this case, the wear by cutting should be the smallest. The wear of the enamel in contact with the Filtek TM Supreme material is the smallest during friction under the two loads applied, amounting to 0.00014 and 0.00021 mm^3/m, respectively. In this composite, the filler content is the lowest and amounts to 55.6%. The particle size in this material is particularly varied because it occurs in two size ranges: 5–75 nm and 0.6–10 μm. It can be assumed that the large, oval filler particles embedded in the 5-component resin, which represents 50% of the material's content, do not cause as much wear as the sharp-edged particles.

The results of nanoindentation tests (Figs. 4–8) indicate differences in values depending on changes in the set parameters. It turns out that the hardness and Young's modulus of the human glaze differ significantly in the values obtained from areas subjected to friction from those obtained from areas without tribological interactions.

In analyzing the obtained results (Figs. 9–13), it may be significant to refer to the study by Arseculeratne et al. [3] and Galo et al. [7], where nanoindentation of surfaces after friction as performed at much higher loads (150 and 200 mN) [3] than those used in this study and with a much shorter test time of the tribological experiment – 30 minutes (for comparison: 3 hours in this article). In the experiment of the authors mentioned above, load was applied within the range of 2–10 N during the friction process, i.e., half of the load in the experiment described in this article. Analysis of the data obtained by the researchers [3], [7] shows that the extent of the change in the properties of the newly formed enamel surface layer after friction ends within the depth reached by indentation, and the corresponding changes are so small as to be statistically insignificant. It can be assumed that the surface modification resulting from the tribological process extended to a very low depth, and during penetration, the indenter penetrated to a depth beyond this range of changes, showing the hardness value and the Young's modulus of enamel unchanged by friction.

In our experiment, the obtained data suggests the formation of a new modified layer within the contact depth range of about 100–700 nm (Figs. 9–13), where changes in the values of the tested strength parameters still occur. So it may be that we have not yet reached the depth of the enamel layer untouched by tribological processes. The depth of 700 nm is still a depth within the tribological response of dental enamel.

For some materials, the tendency of alignment of mechanical parameters (H and E) at the highest preset loading of the indenter is observed. This may indicate that we have nearly reached the maximum depth range of the modified layer on the surface of the enamel after friction. The indenter penetrated into the material at a maximum load of 40 mN up to a depth of about 700 nm. This depth, where changes in the values of mechanical properties appear, testifies to the thickness of the surface layer of enamel modified by the friction process.

It is significant that the hardness of the enamel (without friction) ranges from 4000 MPa to 9500 MPa. This may mean that the individual characteristics of human enamel differentiate its properties. It is therefore important to compare parameters between places subjected to friction and places not subjected to friction. Then the analysis of results will be more reliable.

The nanoindentation measurement results showed some changes in the mechanical properties of the surface of tooth layers that occurred after the wear test. This is clearly visible for every tribological pairs. Differences in hardness values are observed on the surface subjected to friction under a load of 23.5 N depending on the penetration depth of the indenter. These differences are less visible for Artiste and Filtek TM Supreme. For Boston and Super Cor, this is an upward trend, and a downward trend for the other materials. For a surface changed by friction under a 7.7 N load, only Artiste exhibits an upward trend. Young's modulus values exhibit similar characteristics.

From the graphs above (Figs. 4–13), we can conclude that the enamel surface is more resistant to wear in the area affected by friction than the deeper layers of material. We see a decrease in hardness and Young's modulus as depth increases. This means that the enamel surface layer is modified by the contacting dental material. This is shaped to a certain depth and different thickness ranges of the changed layer have different properties depended from the use different dental materials in tests.

5. Conclusions

The results obtained in the experiment show that each of the materials affected the enamel surface layer differently, and the modification of these layers takes place up to a certain depth depending on the composite used. Poorly mechanically resistant layers, with different properties compared to non-friction enamel, are formed. It is assumed that the mechanism of changes in the properties of these layers relates to their transformation into a structure similar to that of a solid lubricant, which may be an answer to the independent variable in friction – the composite used. However, clarification of this phenomenon requires further research and analysis.

Preliminary studies show that different materials undergo different wear in contact with dental enamel and wear down enamel in different ways. It turns out that we still do not have a satisfactory material alternative to enamel. It is believed that the shape of the filler particles and their share in volume are of great significance to the reduction of or increase in enamel wear, which was confirmed in the described experiment by obtained volumetric wear values of the applied test materials.

The results of nanoindendation unequivocally show that the hardness and Young's modulus of human enamel devoid of tribological interactions differ significantly in comparison to values measured in areas where friction occurred. As it turns out, this is only valid for small set loads, i.e., 1 mN and 10 mN, while values of mechanical properties determined under 40 mN start to increase, indicating the modification, i.e., the response of the friction surface layer. This shows that the concentration of changes corresponds to the thickness of the thin layer on the surface, as confirmed by the extent of the indenter's penetration depth. The wear process leads to a reduction in, and sometimes an increase of, both hardness and Young's modulus, however, the decrease in hardness is more pronounced.

The data show that the enamel surface layer modified by the contacting dental material is shaped to a certain depth, and different thickness ranges of the changed layer have different properties.

By analyzing the obtained data, we can conclude that the intensity of wear increases the deeper we enter the enamel layer changed by friction. This is because the interactions of individual dental materials take place at different depths of the enamel surface, thereby influencing the character of the layer's formation and the reaction of enamel to the generation of wear products.

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