EAN 2013
51th conference on experimental stress analysis

Wear behavior of hard dental tissues and restorative materials

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Keywords: hardness, elastic modulus, wear, human enamel, dentin, dental composites

Abstract. Human teeth are exposed to various chemical and mechanical factors. From mechanical point of view it includes attrition, abrasion or their combination. Teeth and dental restorative materials are subjected to normal and shear loads. Therefore the contact-based stresses during mastication and teeth wear are of considerable importance.

In order to study wear behavior of enamel, dentine and two dental restorative composite materials scratch test at various contact conditions was employed. Hardness and elastic modulus were measured using nanoindentation with spherical and pyramidal indenters. Residual wear tracks were observed using laser scanning confocal microscopy.

Introduction

The tribiological aspects of human teeth are very complex issue. Dental tissues as well as dental restorative materials have to withstand a range of imposed loads without failure or loss of their functionality during lifetime.

Human teeth have a unique structure composing of enamel, dentin-enamel junction, dentin and pulp. Unlike the other calcified human structures the tooth enamel is incapable of self-repairing while dentin regeneration capacity is only limited [1]. Thus, good wear-resistance is crucial for both the human teeth and dental restorative materials. Aging and other pathological factors lead to gradual wear of enamel and exposure of superficial dentin and its subsequent wear. Therefore it is important to study wear and failure mechanism of human enamel and underlying dentin.

Tooth wear can be defined as phenomenon that occurs whenever a surface interacts with another surface or with chemically active substances [2], which is manifested in gradual removal of material [3]. From mechanical point of view it includes attrition (tooth-to-tooth contact), abrasion (contact between teeth and food or other abrasive objects or substances), or their combination. Processes of microploughing, microcutting, microcracking and microfatigue can be denoted as the main processes of material removal [2,4].

Tooth enamel is the hardest and stiffest substance in the human body. It is the most mineralized tissue in the body composed of approximately 97% inorganic substances, essentially non-stoichiometric hydroxyapatite, 1% organic material, mainly protein, and 2% water by weight [5]. Dentin, due to less mineral content, is less hard and less fragile in contrast to enamel. By weight, it is composed of 70% mineral hydroxylapatite, 20% organic material and 10% water [6]. Both, enamel and dentin possess a complex hierarchical structure with specific features at nanometre and micrometre scales [7].

Investigating the tribological behavior of dental tissues and contemporary dental composites at relevant scale is necessary to understand the processes and mechanism that influence their damage nucleation and wear. It should be noted that mechanical properties could show a high size-dependent character [8].

Depth sensing indentation (nanoindentation) and scratch test have been adopted as two most common techniques developed for assessment of local mechanical properties at nano and micro scale. Both of them are contact based method where the well-defined probe is pressed into the investigated surface at defined conditions. Nanoindentation is mostly used for assessment of hardness and elastic modulus, while scratch test allows to simulate single asperity contact involved during tooth abrasion in a controlled and reproducible way [9].

The aim of this in vitro study is to investigate wear behavior of hard dental tissues and dental restorative materials using progressive load scratch test and nanoindentation.

Materials and Methods

Mechanical and tribological properties of human enamel, dentine and two light-cured composite resin-based dental restorative materials were evaluated. Filltec Supreme (3M ESPE, St. Paul, MN, USA) is a universal restorative composite composed of combination of silica and zirconia filler bonded in a bis-GMA, UDMA, TEGDMA, and bis-EMA resin mixture. SonicFill (Kerr Corporation, West Collins Orange, CA, USA) consists of SiO_2, glass, oxide and other chemical fillers bonded in resin mixture of BisphenolA-bis-(2-hydroxy-3-mehacryloxypropyl)ether, 3-trimethoxysilylpropyl methacrylate, Ethoxylated-bisphenol-A-fimethacrylate, Triethyleneglycoldimethacrylate.

Two healthy human molar teeth extracted for orthodontic reasons were used in this study. After disinfection, the cavity with depth about 5 mm in depth was prepared and filled with the investigated restorative composite. Then the roots were removed from cervical region and slides, along the crown-root direction with thickness of 1 mm, were cut with a diamond saw using water cooling to avoid any overheating. Slides, consisting of enamel, dentin and used dental restorative material, were grid with 320 and 600-grit silicon-carbide and subsequently polished in 0.25 μm diamond suspension. The finishing procedures were also performed under the conditions of water cooling to prevent from overheating.

Scratch tests were performed using a fully calibrated NanoTest instrument with a sphero-conical diamond indenter with 10 μm nominal end radius at room temperature. The actual indenter end radius of 8.0 μm was determined using laser scanning confocal microscope and verified by nanoindentation on standard fused silica sample.

Progressive nanoscratch testing was performed to load of 30 and 200 mN as three scan experiment (topography – scratch – topography). During the scratch procedure the initially constant topographic load of 0.1 mN was applied over the first 50 μm and then ramped to 30 and 200 mN at constant loading rate of 0.78 and 5.2 mN/s, respectively. The initial and final topography was performed over the whole scratch length before and after scratch procedure. All the scans were performed at scan speed of 10 μm/s over total scan length of 450 μm. The topography load of 0.1 mN was sufficiently low in order to avoid any wear during the topography scans. Evaluation of the scratch tests was performed on the basis of the indenter on-load and depth record and analysis of the residual scratch tracks. Laser scanning confocal microscope LEXT OLS 3100 was used for high-resolution imaging.

Spherical indentation with the same probe was carried out to assess hardness and elastic modulus of the investigated samples. These load-partial unload experiments were performed in ten steps at peak loads from 10 to 100 mN with loading and unloading rates set to 1 - 10 mN/s. A 60 s hold period at maximum load was applied before unloading and a 60 s hold period at 90% unloading for correction of any thermal drift. The indentation curves were analysed using the method proposed by Field and Swain [10].

Besides, nanoindentation with sharp Berkovich indenter at maximum load of 50 mN was performed. The standard analysis based on fitting of unloading curve by the power law fit was performed [11]. These data were also used for calculation of total work W_t and reversible elastic work W_e done during the indentation cycle defined as the area under the loading and unloading curve, respectively. Obviously $W_e = W_t - W_p$, where W_p denotes irreversible plastic work done during indentation. The ratio of W_p/W_t is also known as the plasticity index.

Results

Hardness and elastic modulus measured with spherical and pyramidal Berkovich indenter are presented in Table 1. The scratch hardness, also shown in Table 1, is defined by the formula

$$H_s = \frac{4F}{\pi w}, \tag{1}$$

where w is scratch width at a given normal load F as proposed in [12]. The presented average values are calculated for loads of 155, 175 and 190 mN. Nanoindentation with spherical and pyramidal indenter gives almost identical values, while scratch hardness gives slightly higher values. This means that concept of scratch hardness can be used for reasonable estimation of material hardness. It should be noted that calculation of scratch hardness is based on evaluation of dimensions of residual wear track after total unloading, while nanoindentation hardness is calculated with projected area at applied load.

Reduced moduli of dentin and enamel, determined by Berkovich and spherical indenter, are also very similar and within the range of results from other micro- and nano-indentation experiments [13]. Dentin is almost as hard as SonicFill and Filtek Supreme, while its elastic modulus is approximately 30-40 % higher. Enamel is almost four-times harder and approximately three-times stiffer in comparison to dentin.

Table.1 Comparison of scratch and nanoindentation hardness and modulus.

Sample	Hardness *H* [GPa]			Reduced Modulus *E* [GPa]	
	Scratch test	Berkovich	Sphere	Berkovich	Sphere
Dentin	1.2 ± 0.1	1.1 ± 0.2	0.9 ± 0.1	28.6 ± 4.1	25.6 ± 1.3
Enamel	4.1 ± 0.2	4.2 ± 0.3	3.9 ± 0.3	91.3 ± 3.4	85.4 ± 8.9
SonicFill	1.3 ± 0.3	1.0 ± 0.2	0.9 ± 0.0	19.7 ± 1.8	16.6 ± 0.2
Filtek Supreme	2.0 ± 0.2	1.1 ± 0.1	1.0 ± 0.1	17.0 ± 0.7	17.1 ± 0.4

The wear resistance of thin films and coatings has long been considered to be directly defined by their hardness [14]. However, it has been proved, that instead of hardness alone, the ratios of hardness to modulus H/E and H^3/E^2 can be considered as a more reliable parameter for controlling wear of the material [15]. Modern theories of wear relate parameters H/E to the elastic strain to failure and H^3/E^2 to the resistance to plastic deformation [15]. Their values for the investigated samples are summarized in Table.2.

The on-load scratch depth data and topography data for the progressive scratch test performed at 200 mN are shown in Fig.1. The correction for instrument frame compliance and the sample slope were used in all cases. Dentin shows the highest on-load and residual wear track depths. Both dental restorative materials show on-load depth similar to dentine, while their residual scratch depth is rather similar to the depth of enamel. The lowest depths are observed on enamel. The same trends were observed for the scratch test done up to 30 mN. Comparison of Fig.1 and Table 2 clearly implies correlation between H^3/E^2, plasticity index and on-load scratch depth. Correlation is also observed for *H*/*E* and extent of elastic scratch recovery (scratch recovery = [on-load depth – residual depth]/on-load depth [16]). The scratch recovery calculated at 155 mN is about 75% for investigated restorative materials, while it is 45 % for dentin and 55 % for enamel.

Table.2 Summary of parameters calculated from nanoindentation results (Berkovich indenter).

Sample	H/E ratio	H^3/E^2 ratio	Plasticity index
Dentin	0.039 ± 0.012	0.0017 ± 0.0004	0.74 ± 0.17
Enamel	0.046 ± 0.005	0.0088 ± 0.0011	0.70 ± 0.09
SonicFill	0.053 ± 0.016	0.0030 ± 0.0013	0.68 ± 0.10
Filtek Supreme	0.063 ± 0.007	0.0042 ± 0.0006	0.61 ± 0.05

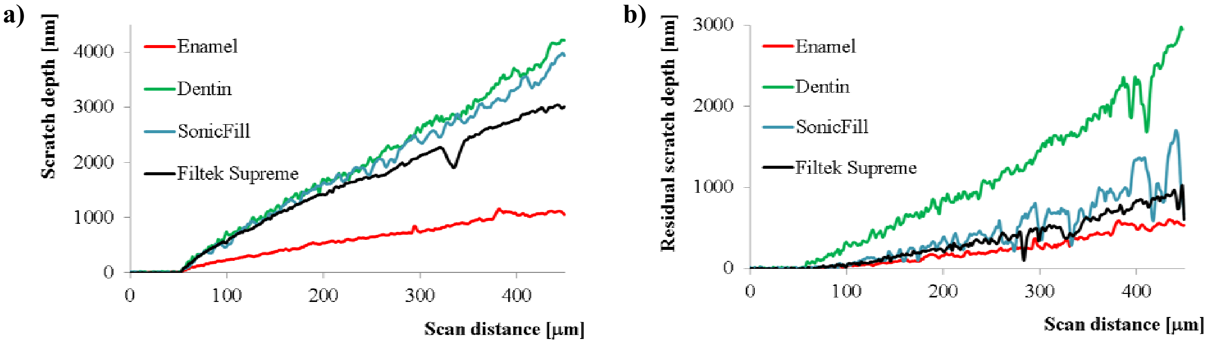

Fig.1 Variation in a) on-load depth and b) residual scratch depth with increasing load.

The wear tracks for 30 mN scratches were too shallow for microscopic observations with maximum depths up to 100 nm, except dentin sample with 400 nm. Representative images of end parts of scratch tracks for all the investigated samples performed at maximum load of 200 mN are shown in Fig.2.

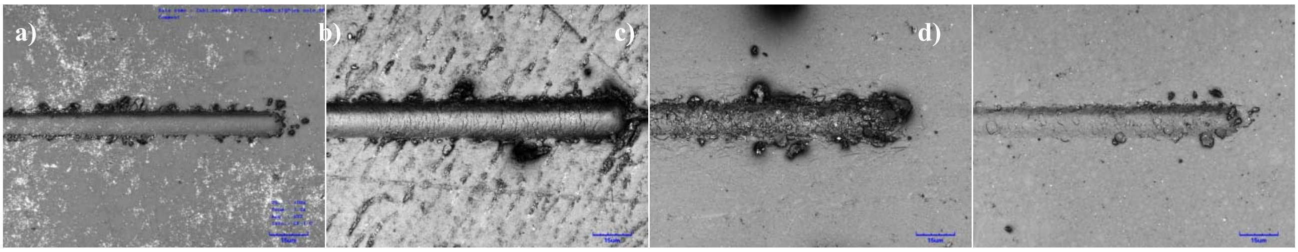

Fig.2 Confocal microscopy micrographs of residual wear tracks a) enamel b) dentin c) SonicFill d) Filtek Supreme.

The worn surface morphologies of all the samples are dominated by plastic deformation at low loads without any traces of cracking. With increasing load, formation of pile up around the wear track occurs. Apparent worn particle packing is observed at the sides of the wear track of enamel and dentin. Detailed analyses of micrographs revealed cracking inside the wear track. The critical load for onset of this cracking is 188 and 157 mN for enamel and dentin, respectively. The morphology of a wear track of Filtek Supreme is almost smooth in contrast to SonicFill with distinct traces of detachment of filler particles from the resin matrix. This can also be seen from the residual wear track profile in Fig.1. The higher susceptibility of SonicFill to detachment at the filler boundary can be linked to the irregular shape of fillers leading to higher stress concentration during the scratch test. On the other hand Filtek Supreme is composed of spherical fillers and exhibits only faint traces of detachment. The onset of fillers detachment was observed at normal load of 60 and 149 mN for SonicFill and Filtek Supreme, respectively. The preliminary experiments indicate that acoustic emission generated during scratch test could give more additional information on deformation mechanism.

The typical scratch profiles at loads of 155 and 190 mN are illustrated in Fig.3. At the same load, the residual depth and width of the wear track can be written with the following equalities: dentin (highest depth and width) > SonicFill > Filtek Supreme > enamel (lowest depth and width).

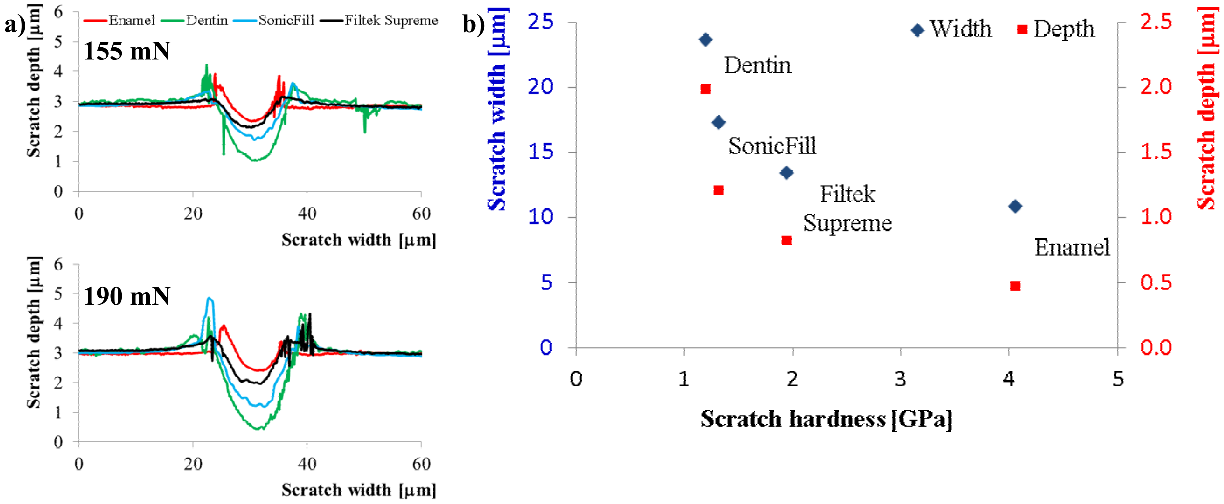

Fig.3. Analyses of residual wear tracks a) surface profiles for 155 and 190 mN b) scratch depth and width as a function of scratch hardness for 190 mN.

Summary

Mechanical properties and wear of human enamel, dentin and two composite restorative materials were measured. Nanoindentation with spherical and pyramidal indenters was performed for assessment of hardness and reduced modulus. Progressive scratch test was used for evaluation of scratch resistance. Both methods have been proved as valuable methods for evaluation of mechanical and tribological properties of dental hard tissues and dental restorative composites at nano and micro scale.

The hardness and reduced modulus were almost identical regardless of indenter geometry. Scratch hardness calculated from dimensions of residual wear track was in reasonable agreement with nanoindentation hardness. The microtribological behavior of the investigated samples differed depending on the microstructure. The scratch recovery correlated with mechanical parameters determined from nanoindentation. Especially, the H^3/E^2 ratio proved to be a suitable parameter for ranking of scratch resistance.

Acknowledgement

This work has been supported by the Technology Agency of the Czech Republic, Grant No. TA03010743.

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