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Indentation Hardness and Tribological Wear under the Conditions of Sliding Friction of the Surface Layer of Composites Based on Methacrylate Resins with Ceramic Nanofiller

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ABSTRACT

The biomaterials, which are the subject of this work, are the dental restorative materials classified as light cured polymer matrix ceramic composites or resin based composites. The dental composite materials are needed for the repairment of human teeth. Fillings and other dental applications are exposed to the biomechanical loading in the chewing process. The wear resistance and hardness are important functional properties. Currently, nanofiller composites play an important role among dental composites. The objective of this paper was to study and analyze the friction, mechanical and wear properties of the surface of polymer matrix ceramic filed nanocomposites. Three material groups were used, one commercial composite Filek Z550 (3M ESPE, USA) and two experimental composites Ex-nano(G) and Ex-flow(G). The microindentation tests were conducted on the Micro Combi Tester device (Anton Paar GmbH, Germany). Rotating sliding ball-on-disc friction tests were performed against an alumina ball on 5 N load at 1 Hz in the bath of artificial saliva at 37°C. The linear wear and friction coefficients were evaluated. In the light of the obtained results of contact and friction strength tests, it was found that the performance depends on the production technology of the polymer-ceramic composites. The test results indicate that the share of filler nanoparticles the in experimental composites is advantageous due to the tribological wear.

Keywords: nanocomposites, polymer matrix ceramic composites, indentation hardness, wear

INTRODUCTION

At present, the composite materials play a key role in biomedical applications, aviation industry, automotive and other engineering applications as they show very good properties [3]. Composites are complex materials that contain at least two chemically distinct phases [7]. These phases are clearly separated and the connection of phases occurs at the macroscopic level [16]. The matrix of the composite is a continuous phase, for the polymer composites these are: thermo-, light- and chemosetting resins as well as thermoplastic resins. The reinforcing phase (filler) usually involves the additives that shape the performance properties. The composite, which formed from the combination of phases, has different properties than that obtained by the components separately. The dispersion reinforcement composites are often reinforced by particles (inclusions) and fibers, also natural ones [4, 16, 39]. The performance characteristics depend on the material structure. Among them, the mechanical and tribological characteristics of the surface layer are important. They can be determined experimentally. The indentation method for assessing the mechanical properties of the material surfaces is relatively easy to conduct. The development of an apparatus for indentation tests makes it a useful tool for various measuring systems of different scale (from macro to nano) [15, 17, 28]. The modern apparatus allows for the measurements in the indenter's load range from kilo-Netwons to pico-Newtons and the measurement of the indenter depth in the surface of the

tested material can be performed with accuracy to nanometers. In addition, the indentation method enables the testing of loose and biological materials as well as nanostructures [17, 31].

Abrasion resistance is an equally important functional characteristic of the material that is caused by friction. There are two types of friction, namely: stiction, which is developed to the highest degree at the moment preceding the motion (impending motion) and kinetic friction – after the start of relative motion [9]. The second type of friction is of major importance for the biomaterials that are used to repair the human teeth. From the basics of friction physics, it is known that the contact of cooperating surfaces occurs at the points of elevation of the roughness profile and the actual contact surface is a small fraction of the nominal contact surface [4]. Polymer dental composites are filled with ceramic particles. The dispersed phase of the composite consists of the particles the three spatial dimensions of which are similar. Due to their shape, the reinforcement in this form is sometimes referred to as 3D reinforcement [23]. The filler usually consists of quartz compounds, ground glass, ceramics and silicon dioxide. The size of the filler particles varies, the size of the pre-polymerized particles as a result of technological process is 1–200 μm, smaller particle agglomerates (1–25 μm), spheroidal fillers (20–30 μm) and nanofillers (<100 nm). The particles of the inorganic phase of polymer-ceramic composites usually contain 70–80% of glass-based filler and 20–30% of nanofillers [32]. The zirconium, titanium and aluminum oxide particles of 250–500 nm are often added. It is obvious that these particles occur on the friction surface and determine the real surface of the bodies in contact and affect the kinetic friction course.

The biomechanics of the chewing process govern the contact of frictional surfaces. Food particles are squeezed and crushed between the opposing teeth and then crushed in the tooth-food-tooth contact, which means that there is friction with the participation of three bodies. In the biomechanical process of chewing, there is also a frictional contact of the tooth with the opposing tooth, in the chewing process, in which the tooth penetrates the food particles completely [Delong 2006]. During normal chewing, the occlusive forces are in the range of 5–20 N and the shape of the curve force – time is close to the positive part of the sinusoidal curve [35]. The tooth speed is from 0.25 to 0.5 mm/s and the chewing frequency is in the range of 1–1.5 Hz [30]. The factors of the oral environment also play an important role for the biotribological behavior; therefore, the tribological tests are also conducted in artificial saliva at a temperature close to physiological one (~37°C) [29]. It follows from the above that the biomechanical loads and physiological environment factors are known. Modern tribometers enable mapping of most of the loads and real factors in laboratory tests. In addition, these methods, as standardized [5,6], allow for the comparison of the results. However, the open data on the mechanical and tribological properties of the dental biomaterials are limited [19]. These properties are important for a clinical success [20]. Therefore, the aim of the research was to gain knowledge about the properties of the surface layer of modern dental composite nanomaterials and conduct a comparative study on these materials.

MATERIAL AND METHOD

Testing materials

Three materials were selected for testing: one commercial (Filtek Z550) [40] and two experimental ones (Ex-nano (G), Ex-flow (G)). The materials are described in Table 1.

The specimens were prepared in accordance with the manufacturers' recommendations. This means that the specimens were shaped by a single operator in a metal split-mold and then light-cured using Megalux LED (Megadenta, Radeberg, Germany) 1200 mW/cm^2 for 40 s with a soft-start system. After photopolymerization, the specimens were polished with abrasive discs (600, 1200, 2400) on a single wheel grinder and polisher Saphir 550 (ATM Gmbh, Mammelzen, Germany) equipment and then cleaned in water. All of the tested specimens were immersed in artificial saliva at 37°C for 30 days to simulate the aging process.

Indentation hardness test

The indentation hardness and modulus of elasticity of the surface were determined according to the Oliver-Pharr method [27]. The test was conducted on a Micro Combi Tester device (Anton Paar). The method is described in more detail in [38]. For the calculation of the modulus

Table 1. Details of tested composites

Material	Manufacturer	Composite type	Matrix (resin)	Filler type	Filler content (wt.%)
Filtek Z550 (abbrev. Z550)	3M ESPE (USA)	Nanohybrid composite	BIS-GMA, UDMA, BIS-EMA, PEGDMA, TEGDMA	SiO_2 20 nm, ZrO_2/SiO_2 5–20 nm nanoparticles, (0.6–1.4 m clusters)	82%
Ex-nano(G)	–	Nano composite	BIS-GMA, UDMA, TEGDMA	The inorganic filler particles consist of barium aluminum, bore glass and highly dispersed silicon dioxide, nanoparticles	82%
Ex-Flow(G)	–	Semi-liquid composite	BIS-GMA, UDMA, TEGDMA	The inorganic filler particles comprise silica, dental glass (strontium aluminum-boron-silicate glass), nanoparticles	74%

of elasticity of the surface E_{IT}, stiffness was determined from the formula (1) [27]:

$$S = \frac{dP}{dh} = \beta \cdot (2/\sqrt{\pi}) \cdot E^* \cdot \sqrt{A} \quad (1)$$

The relationship $\frac{dP}{dh}$ was determined from the graph force – indenter displacement (Fig. 2). In the equation (1), the parameter β for the Vickers indenter is 1.0055 [27]. The magnitude A is a function of the depth h_c (Fig. 2) and it is determined on the basis of the following relationship [18]:

$$A = F(h_c) = 24{,}54h_c^2 + C_1 h_c^1 + C_2 h_c^{1/2} + \\ + C_3 h_c^{1/4} + C_4 h_c^{1/8} + \cdots + C_n h_c^{1/2n} \quad (2)$$

In equation (2), the calculations are based on the constant C_n, which expresses the indenter's geometry. The method for determining the constant C_n is described in the paper [33]. The stiffness calculations include the modulus of the tested surface denoted with the letter *E*. This value is defined by the equation (3):

$$\frac{1}{E^*} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \quad (3)$$

In the equation (3), the value E^* denotes the reduced modulus of elasticity and ν – Poisson's ratio, while E_i and ν_i refer to the indenter. Having calculated the stiffness *S* from the equation (1) and the penetration path of the indenter corresponding to the elastic deformations of the test surface – h_c, the E_{IT} parameter (*E* in the formula 3) was calculated from the formula (4):

$$E_{IT} = \frac{\sqrt{\pi} S}{2\beta\sqrt{A \cdot hc}} \quad (4)$$

In some papers, the researchers use the H/E [24] (indention hardness H_{IT} to the elastic modulus of the surface E_{IT} based on [18, 27, 33]). This coefficient combines the ability to the highest possible elastic deformation (low modulus) and the ability to the smallest plastic deformation (high hardness). For many materials, the yield strength σ_Y is related to the hardness H (typical $H \approx 3\sigma_Y$). The relationship H/E expresses"„elastic strain to failure" of materials and elastic resilience of material. The higher the H/E and H^2/E ratios, the greater the wear resistance a material should have [25, 26].

Sliding wear test

The sliding wear tests of the resin based composites were conducted using the microtribometer (CSM Instruments SA, Switzerland). The ball–on–disk method was conducted. In the wear tests, a ϕ6 mm Al_2O_3 ball counterspecimen was used**.** The parameters of the test: linear speed – 18.8 mm/s, frequency – 1 Hz, normal load – 5N, friction path – 300 m, temperature 37°C, medium (bath) – artificial saliva. The number of specimens in each group was 5 (N = 5). In this present study, the wear was measured as penetration depth of counterspecimen in a composite specimen. The hardness of the counterspecimen was 2000 HV, therefore the ball wear is not evaluated.

RESULTS AND DISCUSSION

Indentation hardness

Table 1 summarizes the results of indentation hardness tests. Mean values, standard deviation (Std.Dev.), minimum (Min) and maximum

(Max) values, sample size (N) and median values are shown. Figure 1 shows the mean "load–penetration depth" indentation curves of all tested materials.

The examination of the indentation hardness according to the Oliver&Pharr method is based on the registration of normal load and penetration depth in real time [27]. The indentation hardness depends on elastic and plastic deformations. The Z550 material has the highest indentation hardness (H_{IT}). The lowest average value of this mechanical quantity was found in Ex-flow (G) material. A similar relationship was demonstrated for the modulus of elasticity (E_{IT}). Both tested conventional composites with higher filler content were harder and had a higher modulus of elasticity of the surface. The dependence of hardness on the filler content was shown in a number of papers [10, 21]. A correlation between indentation hardness, modulus of elasticity and the filler content was shown in the paper [15]. Linear regression confirmed a positive correlation between elastic moduli and filler loading (coefficient of determination $R^2 = 0.98$), and between indentation hardness and filler content ($R^2 = 0.99$) [15]. In the paper [15] similar, but not the same, materials were tested. The results of elastic modulus and indentation hardness tests presented in that paper, for similar composites, are similar but slightly higher. However, the hardness tests in that paper, in contrast to those presented here, were conducted with the use of instrumented nanoindentation method, and the samples were stored in distilled water at 37ºC for 7 days and the light-cured equipment was not the same. In another paper [2], the researchers used the nanoindentation method to determine the hardness and modulus of elasticity of the Filtek Supreme nanocomposite and they were 0.78±0.17 GPa (H_{IT}) and 15.71±1.69 GPa (E_{IT}), respectively, which is close to the values obtained for Ex-nano (g) nanocomposite. According to [8], the use of nanoparticles as part of the filler increases the hardness of the material, the Ex-nano(G) experimental composites contain more nanofiller than Z550. It is possible that the influence of the type and size of filler particles is decisive, yet the type of the dispersed phase is also important, which is different in the tested composites (Table) 1). It is possible that the content of the zirconium particles is important. The studies presented in [14, 36] showed that the hardness of the composite increases along with the content of zirconium particles in the material. The hardness of zirconium particles is 17 GPa, while other popular filler based on silicon compounds, also used in experimental materials, is about 3–4 GPa [21]. Among the composites tested in this paper, Z550 contains the zirconium oxide particles, which may translate into the highest hardness of this material among those tested.

In some papers, the researchers used the H/E ratio [24] (hardness to modulus of elasticity). This coefficient combines the ability to the highest possible elastic deformation (low modulus) and the

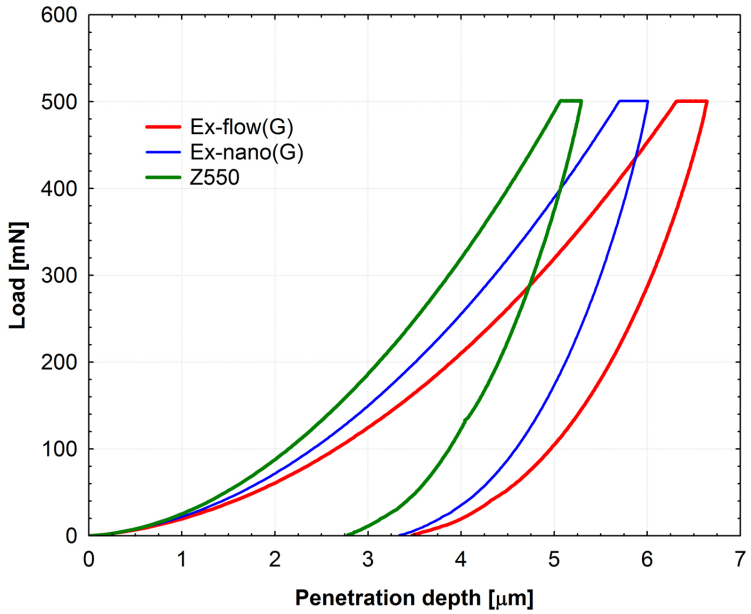

Fig. 1. Mean indentation characteristic (normal load – penetration depth of Vickers indenter)

Table 2. Descriptive statistics of indentation hardness test results

Indentation parameters	Statistic parameters	Ex-flow(G)	Ex-nano(G)	Z550
H_{IT} [MPa]	Mean	602.034	726.416	984.372
	Std. Dev.	36.807	43.242	83.618
	Min.	534.374	673.734	815.113
	Max.	654.48	784.703	1097.508
	N	10	10	10
	Median	609.064	713.7	1018.041
HV_{IT} [Vickers]	Mean	56.824	68.563	92.911
	Std. Dev.	3.474	4.081	7.892
	Min.	50.437	63.591	76.935
	Max.	61.774	74.065	103.589
	N	10	10	10
	Median	57.487	67.363	96.089
E_{IT} [GPa]	Mean	11.603	14.686	16.588
	Std. Dev.	0.501	0.615	0.792
	Min.	10.446	13.753	14.466
	Max.	12.25	15.544	17.672
	N	10	10	10
	Median	11.639	14.522	16.764

ability to the smallest plastic deformations (high hardness). The ratio of hardness to modulus of elasticity (denoted in the literature as H/E, and in this paper, according to the adopted denotations, refers to the variables denoted as H_{IT}/E_{IT}) indirectly determines wear resistance [25]. The following values of this relationship ($H/E \times 10^3$) were obtained: Ex-flow(G) – 51.89, Ex-nano(G) – 49.46, Z550 – 59.34. The lowest value was obtained for the conventional Ex-nano(G) resin nanocomposite. The Z550 material had the highest value. As it turns out, there is no correlation between the indentation hardness (H_{IT}) and the H/E relationship of experimental materials. The hardness of the composite with a higher content of Ex-nano (G) filler is clearly higher than the hardness of the Ex-flow (G) type composite, and the relationship between the H/E ratio of both materials is inverse. The hardness of the conventional Ex-nano(G) composite is 20.66% higher than Ex-flow (G) and the modulus of elasticity is 26.57%. This is why this ratio is slightly more beneficial for the flow material.

Sliding wear

Figure 2 presents the box-whiskers of linear wear of the tested composites. The average wear values vary and depend on the material. The Z550 material had the highest average wear. The wear of the experimental materials was similar. The Mann-Whitney statistical test does not reveal any statistically significant differences between the use of experimental materials ($p = 0.7540$). However, the average value of material wear Ex-flow(G) is lower (similarly as H/E ratio). Other papers revealed smaller wear of flow type composites in the sliding wear tests, but the diversity was greater [29]. This mechanism of increased wear resistance of flow type composites has not yet been explained. The authors of this paper suppose that some particles of the polymer phase of the composite are transferred to the surface of the counter-sample and are applied onto the composite filler particles exposed in the process of wear. According to [1], the transfer film may be in the form of irregular lumps adhering to the smooth counterface, or a continuous uniform layer, which may deform during repeated contact, leading to a smoother counterface.

A friction distance of up to 100 m has an impact on the variability of the friction coefficient of the tested composites. The course of the friction coefficient variability curves in the range up to 100 m is monotonic. In the case of experimental materials, the course of the friction coefficient curves is of stationary character in the range of the friction path 100–300 m. The comparison of the curves presented in Figure 3 indicates that the friction coefficient increases the fastest for the Z550 material, which is reflected in sliding wear (Fig. 2). This material was characterized by the highest wear and coefficient of friction. The average friction coefficient for Z550 was 0.58.

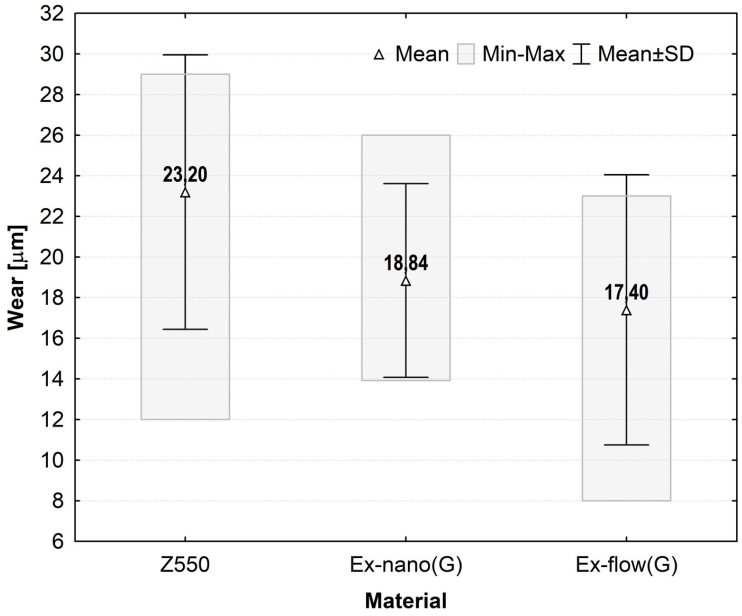

Fig. 2. Box-whiskers chart of linear wear of tested composites

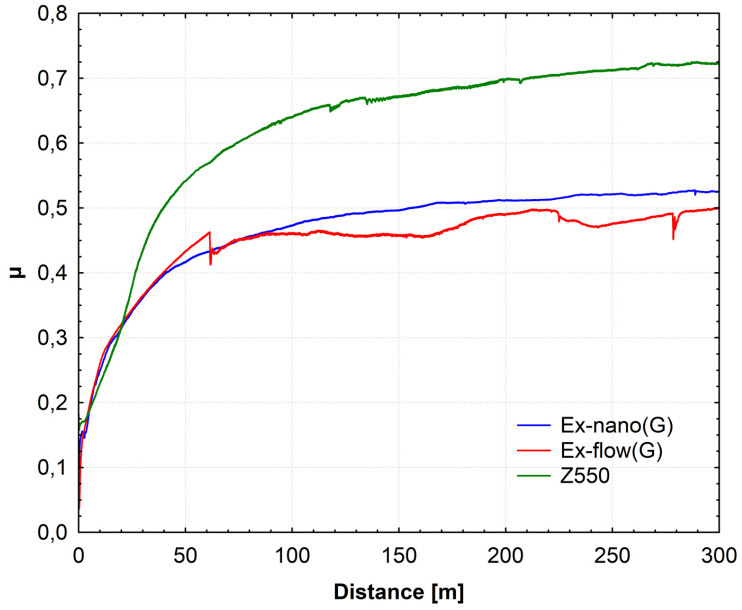

Fig. 3. Representative waveforms of friction coefficient

This means that the average friction coefficient of the experimental materials was ~ 20% lower (Table 3). According to [34], too high values of the friction coefficient may lead to premature wear, while too low ones – to lower efficiency in grinding food.

The differences in the wear resistance, shown in these tests, are consistent with the conclusions described in the paper [37]. The author states that the wear depends on the structure of materials in contact and the geometry of the contacts, the interaction conditions in the kinematic pair – loads, stresses, load duration, surface condition and, finally, the environment as well as the working temperature. In addition, the research showed that the value of the H/E ratio is not correlated with the resistance to the tribological wear. Therefore, for the newly developed polymer-ceramic dental composites, it is not possible to predict the tribological wear resistance based on the mechanical size of the surface layer.

It seems that the higher wear resistance of the experimental composites depends on the proportion of nanofiller particles, which are greater than

Table 3. Descriptive statistic of friction coefficient

Materials	Mean	Minimum	Maximum	Std. Dev.	Coef. Var.
Ex-nano(G)	0.46	0.41	0.51	0.05	9.87
Ex-flow(G)	0.45	0.40	0.48	0.03	7.70
Z550	0.58	0.44	0.66	0.09	14.84

in the commercial Z550. There are hypotheses in the specialist literature explaining the effect of nanoparticles. According to Kleczewska [22], the mechanism is as follows, if the sizes of the filler particles and the space between them are smaller than the strains and deformations caused by contact of two bodies, then the material behaves as homogeneous and its wear resistance is similar to the resin base. If the filler particles and the scale of deformations are similar or the filler particles are larger, the material behaves as heterogeneous one, the consumption is lower than the use of the resin base. In addition, according to [12], the improvement of the properties of surface composites is the effect of high proportion of the filler in the matrix and the small particle size, provided they are well dispersed in the material. Large filler particles increase the friction coefficient and thus the friction forces [12], which may also indicate the beneficial effect of nanofillers used in the structure of experimental composites that were tested.

CONCLUSIONS

On the basis of the literature study and own research, the following conclusions were made:

- In the light of the obtained results of contact and friction strength tests, it was found that the performance depends on the production technology of polymer-ceramic composites.
- The research did not show any correlation between indentation hardness, modulus of elasticity of the surface and the tribological wear. There is also no correlation between the H/E ratio and the tribological wear. Higher hardness does not translate into higher resistance to the tribological wear.
- The research results indicate that the share of the filler nanoparticles in the experimental composites is favorable due to their tribological wear resistance.
- It seems that further development of polymer-ceramic composites is associated with the recognition of the behavior of a given material under the modeling conditions, as precisely as possible, corresponding to the operating conditions.

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