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TRIBOLOGICAL TESTS OF THE NANOMATERIALS USED TO RECONSTRUCT MOLARS AND PREMOLARS WITH THE APPLICATION OF THE DIRECT METHOD

BADANIA TRIBOLOGICZNE NANOMATERIAŁÓW DO ODBUDOWY ZĘBÓW TRZONOWYCH I PRZEDTRZONOWYCH METODĄ BEZPOŚREDNIĄ

Key words:

stomatognathic system, composite, friction, wear, SEM (scanning electron microscopy), nanostructure.

Abstract

Physiological abrasion of teeth is a process of gradual loss of the hard tissue of opposite teeth resulting from age-related natural dental wear. In abrasion, the cusps and their slopes in the jaw and the mandible become flattened due to the mechanical load applied. The aim of this paper is to carry out a tribological and microstructural evaluation of modern composite materials used to reconstruct the teeth in the lateral part of the dental arch. Five light-cured composite materials were selected for tests. The tests involved the coefficient of friction and resistance to wear in a sliding contact in the presence of artificial saliva and the microstructure of the external surface of samples before the wear process and in the wear-related damaged area. The test method applied, which combines a biomechanical analysis of resistance to wear and the analysis of the microstructure before the wear process and in the wear-related damaged area, makes it possible to evaluate the tribological properties of composite materials used to reconstruct teeth in the lateral part of the dental arch.

Słowa kluczowe:

układ stomatognatyczny, kompozyt, tarcie, zużycie, SEM, nanostruktura.

Streszczenie

Fizjologiczne starcie zębów jest to proces stopniowej utraty twardych tkanek zębów przeciwstawnych w wyniku naturalnego zużycia postępującego z wiekiem. Starcie to polega na jednoczesnym spłaszczeniu guzków i ich stoków w zębach szczęki i żuchwy w warunkach obciążeń biomechanicznych. Celem pracy jest ocena tribologiczna i mikrostrukturalna nowoczesnych materiałów kompozytowych stosowanych do odbudowy ubytków w bocznym odcinku łuku zębowego. Do badań wybrano 5 materiałów kompozytowych utwardzanych światłem. Przeprowadzono badania współczynnika tarcia i odporności na zużycie w kontakcie ślizgowym, w obecności sztucznej śliny oraz badania mikrostrukturalne warstwy wierzchniej próbek przed procesem zużycia oraz w strefie skazy zużyciowej. Zastosowana metoda badań łącząca biomechaniczną analizę odporności na zużycie z analizą mikrostruktury przed procesem zużycia oraz w skazy zużyciowej pozwala na ocenę właściwości tribologicznych materiałów kompozytowych do odbudowy zębów w bocznym odcinku łuku.

INTRODUCTION

One of the most important criteria used in the selection of dental materials is their resistance to wear in the contact of the chewing surfaces of the teeth of the opposite arches and the ability to ensure the correct

external surface structure of the filling. In order to ensure correct functioning of the cavities filled, it is necessary to reconstruct the missing tissues in a way that is the closest to natural teeth in terms of biomechanics and the biotribology of occlusion as well as aesthetics. This kind of reconstruction ensures a similar-to-physiological transfer

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of occlusal forces and the replication of the wear process for the reconstructed tissues [L. 1–3]. This subject was selected for discussion, because there are no analyses comparing the tribological properties of the materials used in the direct reconstruction of molars and premolars.

Imitation of natural teeth requires that each tooth of the upper and lower dental arch must contact two opposite teeth, except for the mandibular central incisors and the upper teeth (No. 7), which, as the last in the arch come into contact with a half of the lower tooth (No. 7).

The upper dental arch in the shape of a semi-ellipse covers the mandibular dental arch in the shape of a parabola (Fig. 1). In the coronal plane, the most beneficial contact points are described in literature as ABC (Fig. 2). Point A, located on the internal slope of the buccal cusp of the upper tooth comes into contact with the external slope of the buccal cusp of the lower tooth. Point B, located on the internal slope of the palatal cusp of the upper tooth, comes into occlusal contact with the point located on the internal slope of the buccal cusp of the lower tooth. Point C, located on the palatal cusp, is just like point B, but on its external slope, it comes into contact with the point located on the internal slope of the lingual cusp of the lower tooth. In young people with a complete set of teeth without age-related physiological wear, cusps have an oval shape, so the contact between opposite teeth is achieved not on surfaces but on points. As abrasion of opposite teeth progresses, contact surfaces become larger.

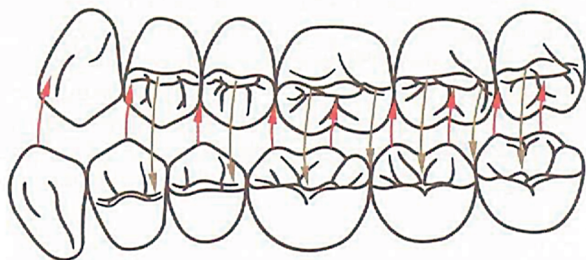


Fig. 1. The representation of correct occlusal contacts following the rule “one-tooth-to-two-teeth.” The arrows indicate contact points between the functional cusps of the teeth of one arch and the enamel laminae and grooves of the teeth of the opposite arch [L. 4]

Rys.1. Schemat prawidłowych kontaktów okluzyjnych według reguły „zęb do dwóch zębów”. Strzałki wskazują punkty kontaktów pomiędzy guzkami funkcjonalnymi zębów jednego łuku a listewkami szkliwnymi i bruzdami zębów łuku przeciwnego [L. 4]

The changes that may sometime occur in the stomatognathic system (SS) during a person's lifetime may become a physiological adjustment standard. When the standard is no longer met, the changes may qualify as pathological dental wear. With regard to teeth, it is an irreversible process of the wear of the hard tissue of teeth that, within the physiological standard, may cause adjustment-related changes in the remaining elements of

SS without resulting in any morphological or functional irregularities. Physiological abrasion of teeth is a process of gradual loss of the hard tissue of opposite teeth as a result of the natural process of age-related wear. In the process of abrasion, cusps and their buccal slopes in lower teeth become flattened along with cusps and their lingual slopes in upper teeth. The process of tooth tissue wear is a product of collaboration between the factors acting jointly or consequently. It is estimated that an average rate of tooth wear in modern humans is 15–20 μm or, according to some sources, 10–40 μm [L. 5, 6].

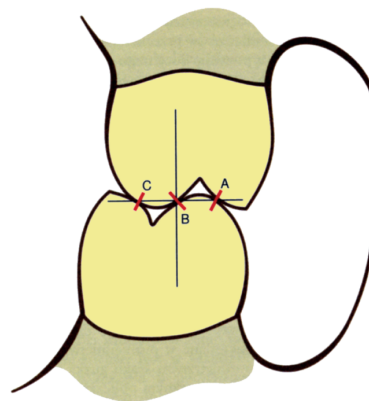


Fig. 2. A representation of correct occlusal contacts ABC in the coronal plane

Rys.2. Schemat prawidłowych kontaktów okluzyjnych ABC w płaszczyźnie czołowej

The aim of this paper is to carry out a tribological and microstructural evaluation of modern composite materials used to reconstruct teeth in the lateral part of the dental arch.

MATERIAL AND METHOD

The materials selected for tests included 5 light-cured composites with very good aesthetic properties. They were based on innovative technologies and may become an alternative for traditional fillings used in teeth that carry great occlusal load. The light-cured materials selected were state-of-the-art products as the time of shaping the filling is long. The following composites were tested:

- Ceram.X Sphere TEC is universal for all classes of fillings in the frontal and lateral part, and the chameleon effect makes it possible to achieve a colour-matching and blending effect. It is resistant to the forces that work in the lateral part and contains nanoceramic filler.
- Rok – SDI is a hybrid composite used in the fillings of the back parts of dental arches, resistant to the chewing forces. Its compressive strength is close to amalgam, and it has low polymerisation shrinkage, with reduced sensitivity to peripheral leaks and high filler contents.

- Solitare – Heraeus Dental is light-cured composite made of polyglass. It makes tight fillings and releases fluoride. It is non-penetrable by X-rays and is used for the fillings in back teeth, because it resistant to chewing forces and wear. It is flexible with a firm texture and enables easy reconstruction of the contact point with opposite teeth.
- Charisma – Heraeus Dental is a universal light-cured composite to be used in front and back tooth fillings. Its aesthetic properties and durability has been clinically confirmed over many years, and has an abrasion similar to natural teeth, and it protects opposite teeth.
- Empress Direct – Ivoclar Vivadent is a universal, non-hybrid material of the highest aesthetic qualities for direct reconstruction of cavities. It is characterised by excellent polishability, opaqueness close to that of natural teeth, and fluorescent and opalescent properties, which are necessary when preparing beautiful and natural-looking fillings.

The samples prepared were in the shape of discs of the diameter of $\varnothing 1/4$ " and the thickness of $1/16$ " ; 15 items made of each material. The preparation methodology was in line with the clinical procedures applied in cavity reconstruction. The discs were made using standard openings of a diameter of $\varnothing 1/4$ ", which were insulated on glass pads. The process of material preparation, application, and curing using a LED lamp was carried out following the producers' recommendations. After curing, the samples were initially processed and then polished. The reference materials were enamel samples prepared as discs from normal premolars and molars. Before the test, the samples were soaked in saline solution for 48 hours.

The tests performed involved testing the coefficient of friction and resistance to wear in a sliding contact in the presence of artificial saliva. Saliva is a medium with large contents of enzymes and plays a very important role in lubricating occlusal surfaces [L. 7–9]. Tribological tests were performed on Roxana Machine Works with the application of the friction node made of a ball and 3 discs of the tested material. The counter samples were ceramic balls $S\varnothing 1/2$ " of zirconium oxide, made with the margin of error of 0.00013mm following ASTM F2094-02a. Microstructural tests of the external surface of samples before the wear process and in the wear-related damaged area were carried out using a Hitachi S-3400N scanning microscope. The samples were first sputtered with gold.

In SS, there is a set of biomechanical extortions caused by external stimuli, such as chewing, mimics, breathing, articulation, as well as internal stimuli, i.e. the performance of mechanical functions controlled and supported by the central nervous system (CVS). The greatest loads in the SS occur during chewing. Chewing involves a complex system of movements that include the rhythmical movement of the mandible up and down (opening and closing) with simultaneous protrusion and

retrusion, rotation in the coronal plane, and the lateral motion of the mandible.

The geometry of the node tested was similar to the arrangement of SS in space (Fig. 3). It was possible to simulate the change in the intensity of wear that was observed in *in vivo* trials and is related to the change of the area of the contact surface. The tests were performed in the following conditions:

- A rotational speed of 200 rev/min ± 10 rev/min,
- A temperature of work $36.6^{\circ}\text{C} \pm 1.5^{\circ}\text{C}$,
- A load of 300 N ± 10 N, and
- A duration of min ± 15 s.

The measure of anti-wear properties of the tested materials was the average diameter of the wear-related damaged area. During the test, the moment of friction was continuously recorded, and, on the basis of these measurements, the coefficient of friction was determined.

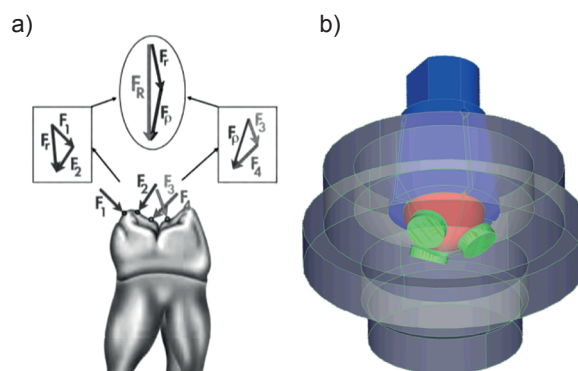


Fig. 3. The spatial distribution of force in occlusal contacts (a), friction node geometry in tribological tests (b)

Rys. 3. Przestrzenny układ sił w kontaktach okluzyjnych (a), geometria węzła tarcia w badaniach tribologicznych (b)

TEST RESULTS AND DISCUSSION

The complex materials currently applied in conservative dentistry – composites – continue to be modified and prepared as materials whose properties are as close as possible to the properties of hard tooth tissues. Optimal resistance parameters are preferred for the state-of-the-art composites to ensure that they can be used to reconstruct teeth in the lateral part of the dental arch where occlusal loads are 3–4 times greater than in the frontal part. Following the test results for the coefficient of friction in the conditions set, the materials may be divided into three groups (Fig. 4):

- The lowest value of the coefficient of friction – 0.15 – was confirmed for CeramX.
- The higher values of the coefficient of friction – 0.55, which is closest to enamel, was confirmed for Empress Direct.
- The highest values of the coefficient of friction – 0.7–0.85 – was confirmed for Solitare, Charisma, and Rok.

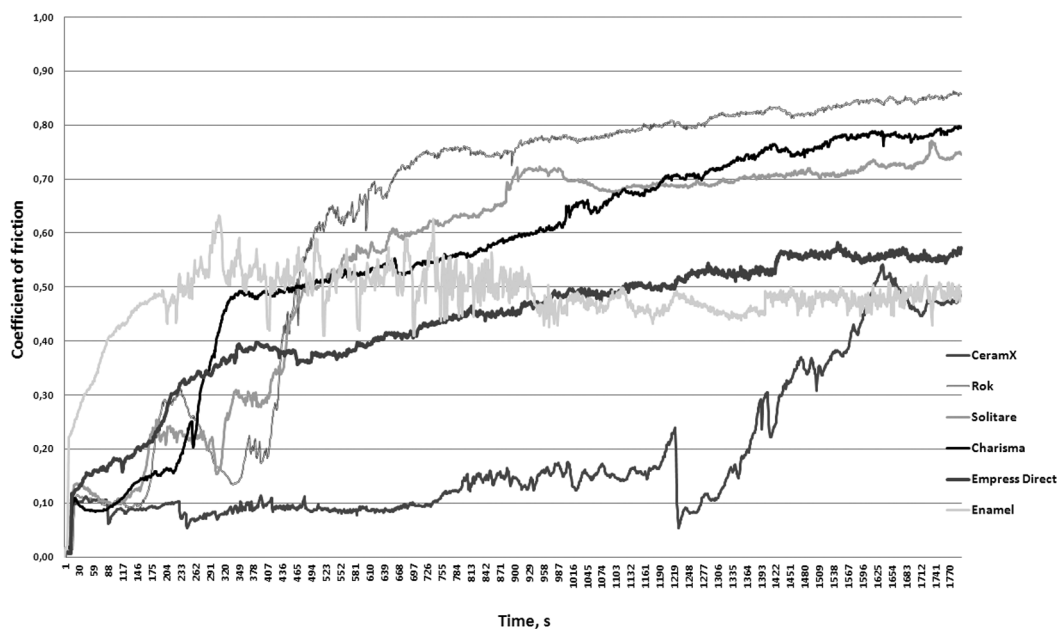


Fig. 4. Representative coefficients of friction for the composite materials and the enamel tested in collaboration with ZrO_2

Rys. 4. Reprezentatywne współczynniki tarcia badanych kompozytów i szkliwa we współpracy z ZrO_2

The evaluation of the coefficient of friction makes it possible to state that CeramX, both at the start of the procedure, as well as during the procedure, was characterised by the lowest stable values of the coefficient of friction. Other composite materials, such as Charisma, Solitare, Empress Direct, and Rok, at the start of the respective procedures had much lower comparable values that stabilised during the procedures at much higher values. The greatest resistance to motion was confirmed for Rok. The closest-to-enamel value of the coefficient of friction during the procedure was confirmed for Empress Direct. The properties of enamel should also be mentioned. Tribological cooperation in the first part of the procedure was accompanied by

vibrations and acoustic effects and the coefficient of friction obtained was at the level of 0.43–0.60. In the second part of the procedure, the coefficient of friction stabilised at the level of 0.48. Earlier enamel tests carried out by the authors indicated high regularity in the spatial image of the external surface and very low values of R_a and R_{MS} parameters characterising this layer (Fig. 5). On the basis of the tests of the internal enamel structure, it may be said that, as a result of the high mineralisation of the enamel tissue and the stochastic distribution of prisms under the external surface, the tribological processes follow a special course after the external surface becomes worn and the internal structure is exposed (Fig. 6) [L. 10–13].

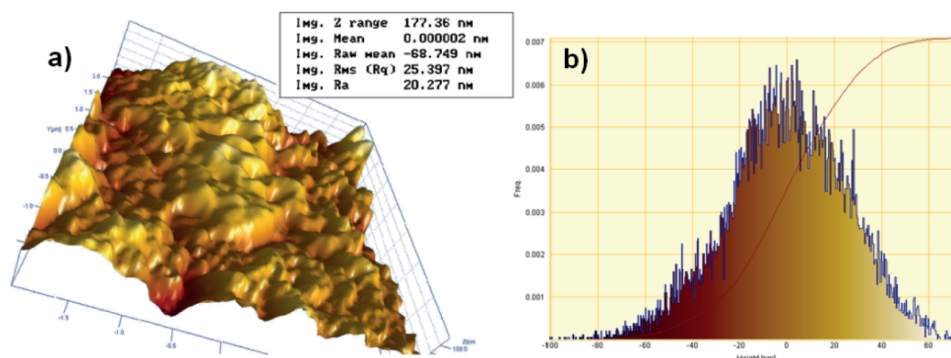


Fig. 5. The results of molar enamel external surface tests: a) spatial image of the correct enamel external surface on the basis of AFM, b) distribution of uneven height values in the correct enamel external surface as a function of the frequency of occurrence

Rys. 5. Wyniki badania warstwy wierzchniej szkliwa trzonowców: a) przestrzenny obraz warstwy wierzchniej szkliwa prawidłowego na podstawie AFM, b) rozkład wartości wysokości nierówności w warstwie wierzchniej szkliwa prawidłowego w funkcji częstotliwości występowania

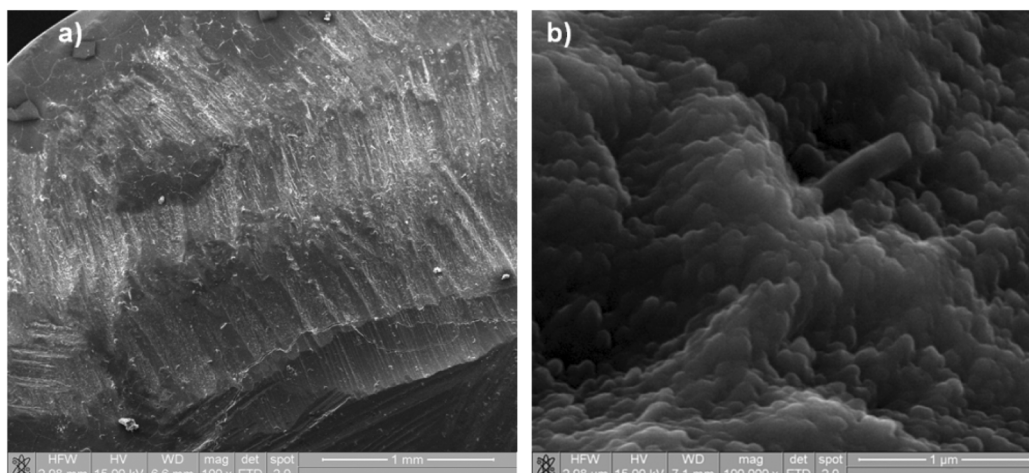


Fig. 6. SEM images of enamel prisms in the internal structure

Rys. 6. Obrazy SEM pryzmatów szkliwnych w strukturze wewnętrznej

In the tests of resistance to wear, the smallest wear-related damaged area was obtained for CeramX – 2.06 mm (**Fig. 7**). It may be concluded that, not only does this material have the smallest resistance in a sliding contact, but also demonstrates the smallest extent of wear. For the second nano-composite, Empress Direct, the wear-related damaged area was 2.35 mm. The remaining three materials, Charisma, Solitare, and Rok, had similar properties in terms of resistance to wear. Wear-related damaged areas for these materials were within the range of 2.56–2.72 mm. If you compare resistance to wear of enamel, which was 3.14 mm, it can be said that, after the reconstruction of cavities with these materials, the ensuing adjusted wear will be at a slightly lower level than for natural tissues. All tested materials may have clinical applications for the reconstruction of cavities in the frontal and lateral parts of the dental arch.

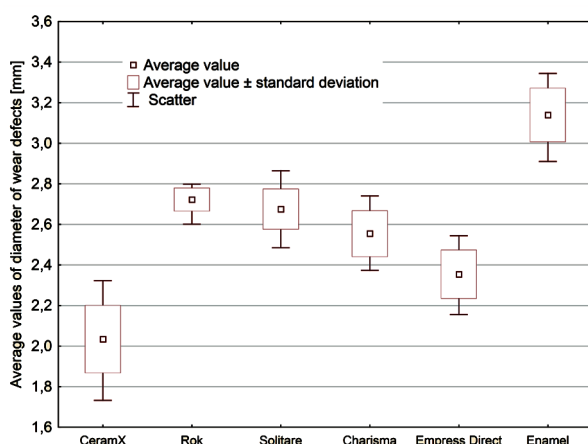


Fig. 7. Wear resistance tests for the materials used to reconstruct teeth and enamel

Rys. 7. Badania odporności na zużycie materiałów do odbudowy zębów oraz szkliwa

Resistance to wear was characterised by the parameters of descriptive statistics: the mean value, the standard deviation, and the dispersion of test results (**Tab.1**). In the next stage, Shapiro-Wilk tests were carried out for the results obtained for each group of materials. All groups of results had a normal distribution (Shapiro-Wilk test $p > 0.05$). At the second stage of statistical analyses, ANOVA tests, which confirmed the existence of significant statistical differences between the materials tested, were executed. In order to identify which differences between materials are important, the post-hoc tests were performed (Fisher's test, Tukey's test). The statistical values were tested at the significance level of $p < 0.05$. Calculations were made using the Statistica 13.1 package.

Depending on the size and volume of filler particles, the materials used in reconstruction dentistry may be divided into composites with macro-fillers, micro-fillers, and nano-fillers. The next generation are hybrid materials that are made by adding filler particles of different size (0.8–5 μm). They may include macro-hybrids, intermediate hybrids, and micro-hybrids. The micro-hybrid materials include Charisma, Rok, and Solitare, whose properties have been confirmed in long-term clinical trials [**L. 4, 13–24**].

In this context, dental composites are understood as materials comprising curable dimethacrylic resins based on hydrocarbon molecular structures and methacrylate functionalised but otherwise non-reactive fillers. Setting occurs due to radical polymerisation of the resins. Compomers as another important subgroup of dental restoratives comprise methacrylate functionalised reactive fillers and polyacid modified methacrylate resins, which promote an additional ionomer setting reaction accompanied by fluoride release.

Microfilled composites comprise only microfillers with an average agglomerate size of $< 0.4 \mu\text{m}$. To increase filler load, microfilled composites contain

Table 1. Tested materials – statistics

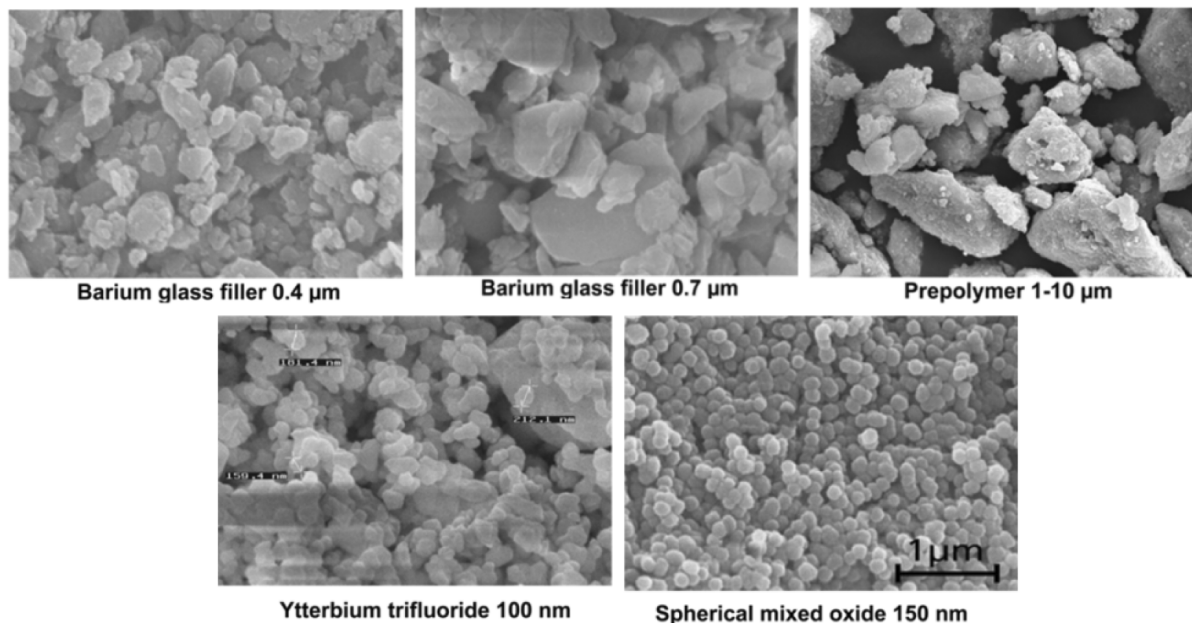
Tabela 1. Testowane materiały – statystyki

Tested material	Statistical parameters for description				
	Number of samples	Mean value	Minimum value	Maximum value	Standard deviation
CeramX	30	2.03	1.73	2.32	0.17
Rok		2.72	2.60	2.80	0.06
Solitare		2.68	2.49	2.87	0.10
Charisma		2.55	2.37	2.74	0.11
Empress Direct		2.35	2.16	2.54	0.12
Enamel		3.14	2.91	3.34	0.13

also prepolymerised microfilled resin. To further increase filler load and mechanical strength, hybrid composites comprise, besides the agglomerates known from microfillers, solid glass fillers instead of the prepolymerized resin particles. These glass fillers are of an average particle size of about 1–10 μm . Recent developments lead to smaller sizes of the glass filler fraction with an average particle size of about 0.4–1 μm resulting in the “micro-hybrid composites.” In general, high filler loads support mechanical strength and reduce polymerisation shrinkage. Larger filler particles

facilitate high filler loads due to their lower specific surface area and the corresponding lower energy to wet these particles with resin. On the other hand, smaller particles are favourable to obtain superior aesthetics, polishability, and wear resistance. The homogeneous dispersion and complete resin wetting of nano-sized filler particles is desired to improve the aesthetic and mechanical properties of composites [L. 25].

The new nano-hybrid material is Empress Direct in which the filler particles used are of diverse size and geometry (Fig. 8).

**Fig. 8. SEM images and the size of filler particles for Empress Direct [L. 26]**

Rys. 8. Obraz SEM oraz wielkości cząstek wypełniacza w materiale Empress Direct [L. 26]

CeramX belongs to a group of the most modern polymer nano-composites (Fig. 9). The smallest particles of the filler are below 100 nm in size. The organically modified ceramic nano-particles comprise a polysiloxane backbone. The chemical nature of the siloxane backbone is similar to that of glass and ceramics. Methacrylic groups are attached to the backbone via silicon-carbon-bonds. These nanoceramic particles can be best described as inorganic-organic hybrid particles,

where the inorganic siloxane part provides strength and the organic methacrylic part makes the particles compatible and polymerisable with the resin matrix. The nanoparticles added in the proportion of 3–5% result in the polymer modification caused by the contact between the polymer matrix and the remaining components. The filler particles are diverse in size and ball-shaped (Fig. 10).

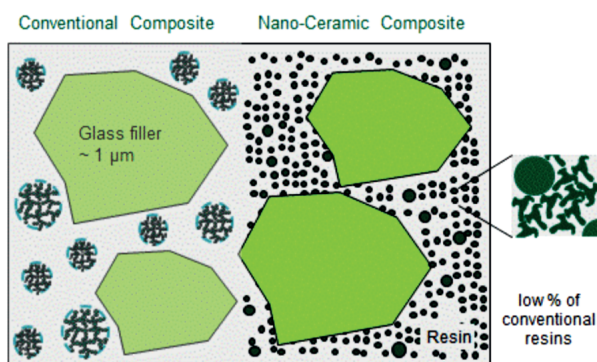


Fig. 9. A representation of CeramX as compared to a conventional composite material [L. 25]

Rys. 9. Schematyczne przedstawienie CeramX w porównaniu z konwencjonalnym kompozytem [L. 25]

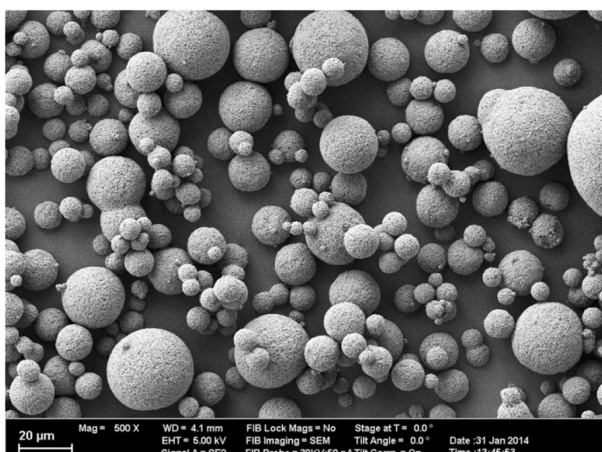


Fig. 10. A SEM image of filler particles in CeramX [L. 27]

Rys. 10. Obraz SEM cząstek wypełniacza w CeramX [L. 27]

The morphological tests of the external surface of the materials tested confirm their structural diversity and are compatible with clinical studies (Figs. 11–15). A comparison of SEM images confirmed the fine-grain regular external surface achieved using CeramX, both at the stage of clinical preparation and after the wear process (Fig. 11). The presence of ball-shaped particles of different nano-size has a very good impact on resistance to wear and on the uniformity of the composite internal structure. No significant morphological differences in the external surface were confirmed before the wear process or in the wear-related damaged area. The results of tribological tests and scanning observations make it possible to claim that this material has very good properties and may be used to reconstruct premolars and molars. Thanks to its biomechanical advantages, it is an optimal material, an alternative for amalgam, and can also be used to reconstruct teeth by milling [L. 22].

SEM images confirm geometric diversity of the nano-hybrid material Empress Direct, whose structure is non-uniform with evidently greater grain than the polymer nano-composite CeramX (Fig. 12). The image is dominated by ball-shaped grains, but elongated grains can also be seen. The structure of the external surface shows a “polishing effect” in the contact with the counter sample, which is characteristic for this material (Fig. 12c). The coefficient of friction is very close the coefficient of friction for enamel and lower than for such composites as Solitare, Charisma, and Rok, while wear is on the same level as for the abovementioned micro-hybrid composites.

SEM images for Solitare show a diversified structure (Fig. 13). Grains differ in size, and some are as large as 10 µm and have an irregular shape. The topography of the wear-induced damaged part indicates polishing of the external surface in a sliding contact but there is also local peeling.

SEM images of Charisma show a diversified structure in terms of the size of grains and chemical composition (Fig. 14). The shape of grains was more irregular than in the nano-composites CeramX and Empress Direct. As compared with ball-shaped filler particles, the particles were more flattened. In the images of wear-related damaged areas, the material was polished while cracks and peeling propagated.

The scanning tests of Rok show a structure with sharp-edge grains, diversified in terms of geometry (Fig. 15). In most of the wear-related damaged area, there are characteristic grooves in the material. The microstructural image confirms the lowest resistance of this material as compared with the microcomposites tested.

When selecting the methodology of the tribological tests of the composites used in the reconstruction of teeth in the lateral part of the dental arch, it is essential to consider the biomechanical and environmental conditions of their work and the morphological evaluation of the external surface before and after wear. The extent of tribological wear is determined by the external surface and the structure of the material obtained in the technological process of composite production as well as the external surface and the structure developed in the process of clinical reconstruction. The process of wear and the reconstruction of the external surface, which occur during the SS adjustment process, are essential. Materials used for reconstruction should be characterised by smaller-than-enamel or similar-to-enamel intensity of wear, the ability to carry large unit pressure, durability, and correct reconstruction of the external surface. In chewing conditions, the external surface is subject to constant wear, which should not impact the opposite occlusal surface.

The statistical preparation of the test results, their repeatability, and the regularities observed confirm the evaluation presented above.

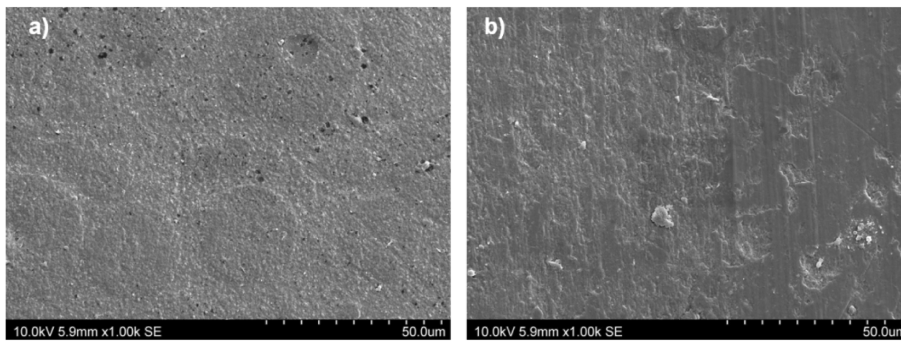


Fig. 11. SEM images of CeramX: a) external surface prepared on the clinical level, b) damaged external surface after the dental wear test

Rys. 11. Obrazy SEM kompozytu CeramX: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w szkacie po badaniu zużyciowym

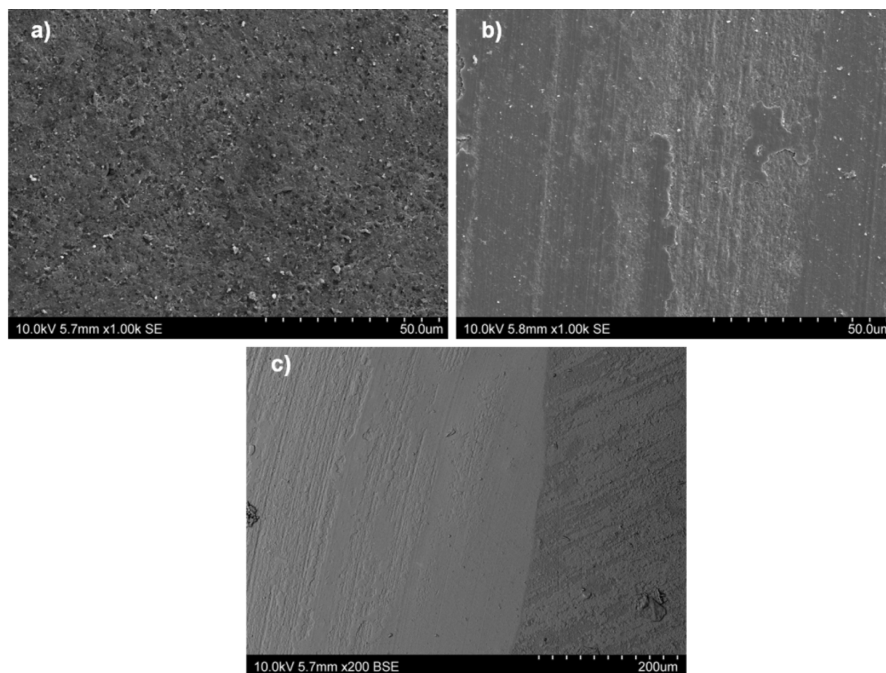


Fig. 12. SEM images of Empress Direct: a) external surface prepared on the clinical level, b) damaged external surface after the dental wear test, c) border zone between the damaged area and the area before the dental wear test

Rys. 12. Obrazy SEM kompozytu Empress Direct: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w szkacie po badaniu zużyciowym, c) strefa granicy między szkazą a obszarem przed badaniem zużyciowym

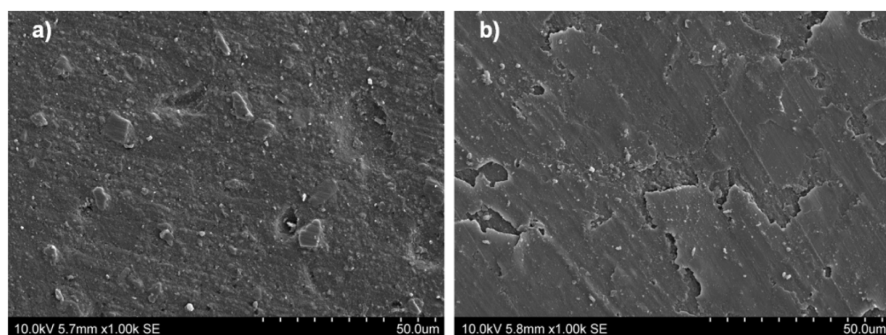


Fig. 13. SEM images of Solitare: a) external surface prepared on the clinical level, b) damaged external surface after the dental wear test

Rys. 13. Obrazy SEM kompozytu Solitare: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w szkacie po badaniu zużyciowym

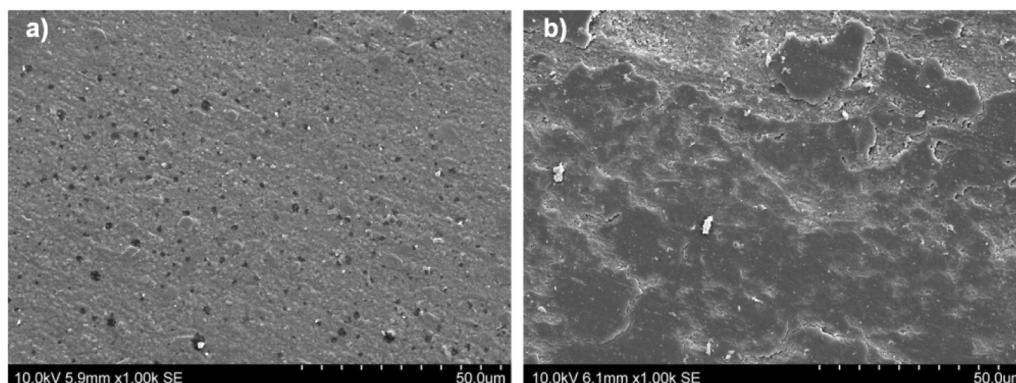


Fig. 14. SEM images of Charisma: a) external surface prepared on the clinical level, b) damaged external surface after the dental wear test

Rys. 14. Obrazy SEM kompozytu Charisma: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w szkiele po badaniu zużyciowym

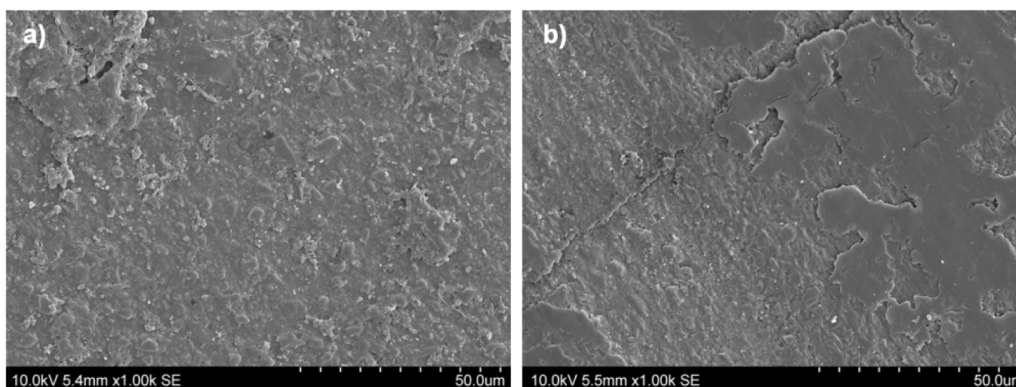


Fig. 15. SEM images of Rok: a) external surface prepared on the clinical level, b) damaged external surface after the dental wear test

Rys. 15. Obrazy SEM kompozytu Rok: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w szkiele po badaniu zużyciowym

CONCLUSIONS

The test method which combines the biomechanical analysis of resistance to wear with the analysis of microstructure before wear and in the wear-related damaged area enables the evaluation of the tribological properties of the composite materials used for teeth reconstruction in the lateral part of the dental arch.

The SEM analysis of the wear-related damaged area enables the identification of the nature of wear for the composite materials tested in the conditions of occlusal load transfer.

The introduction of the nanofiller and the stabilisation of the ball-shaped parts in the hardened structure of the material result in reducing the coefficient of friction and increasing the resistance to tribological wear. The regular, fine-grain nanostructure was confirmed in scanning tests.

Despite the statistically significant difference in resistance to wear, all of the materials tested may have a clinical application in tooth reconstruction, in particular, the reconstruction of premolars and molars.

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