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TRIBOLOGICAL TESTS OF NANOCOMPOSITES FOR DIRECT RESTORATION OF TEETH

BADANIA TRIBOLOGICZNE NANOKOMPOZYTÓW DO ODBUDOWY BEZPOŚREDNIEJ ZĘBÓW

Key words: structure, composite, nanoparticles, coefficient of friction, wear.

Abstract The development of the interphase structure of polymer nanocomposites provides them with tribological properties difficult to obtain in traditional microfiller composites and microhybrid composites. In material solutions, it is preferable to modify the parameters of the material in order to obtain properties that are as close to the hard tissues of the teeth as possible. The aim of the study is to compare the wear resistance of new light-cured nanocomposites and to analyse the surface layer and structure before and after the wear process. Recently developed modern materials were selected for the study. In nanocomposites, G-aenial, G-aenial X FLO, and Essentia, an optimal structure was found in SEM images before and after wear, as well as low resistance to sliding friction and resistance to tribological wear. All tested composites were characterized by suitable properties for clinical applications.

Słowa kluczowe: struktura, kompozyt, nanocząstki, współczynnik tarcia, zużycie.

Streszczenie Rozwinięcie struktury międzyfazowej nanokompozytów polimerowych nadaje im właściwości tribologiczne trudne do uzyskania w tradycyjnych kompozytach z mikrowypełniaczami i kompozytach mikrohybrydowych. W rozwiązaniach materiałowych preferuje się taką modyfikację parametrów tworzywa, aby uzyskać właściwości najbardziej zbliżone do tkanek twardych zębów. Celem pracy jest porównawcza ocena odporności na zużycie nowych nanokompozytów utwardzanych światłem oraz analiza warstwy wierzchniej i struktury przed i po procesie zużycia. Do badań wytypowano nowoczesne materiały, które zostały opracowane w ostatnim czasie. W nanokompozytach G-aenial, G-aenial X FLO i Essentia stwierdzono w obrazach SEM jednorodną strukturę na etapie opracowania klinicznego oraz strukturę pozbawioną wyraźnych zarysowań, złuszczeń i wykruszeń po procesie zużycia, a także małe opory ruchu w tarciu ślizgowym i odporność na zużycie tribologiczne. Wszystkie badane kompozyty charakteryzowały się odpowiednimi właściwościami do zastosowań klinicznych.

INTRODUCTION

Polymer nanocomposites constitute a modern group of materials for conservative direct restoration of hard dental tissues. Technologies related to these materials are dynamically developing. The dispersed phase forms structures below 100 nm in them. The significant development of the interphase structure provides nanocomposites with properties that are difficult to obtain in microfiller composites and microhybrid composites. They exhibit better mechanical and aesthetic properties than conventional composites with the same composition. The introduction of a 3–5% nanoadditive to the polymer matrix is sufficient to modify the composite structure **[L. 1–5]**. Modification of the structure results in the development of the contact surface of the nanoadditive with the components of the composite. The aim of the study is to compare the wear

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resistance of new light-cured nanocomposites and to analyse the surface layer and structure before and after the wear process. Conducting tribological and structural studies of nanocomposites for restoring cavities using the direct method results from a large variety of these materials and from innovative technologies applied in their production. Diversified and even conflicting study results concerning the quality of available materials and the continuous appearance of new proposals on the market make it necessary to keep up-to-date comparative assessments **[L. 6–9]**. They will take into account the results of studies performed for nanocomposites used in teeth restoration in the lateral part of the stomatognathic system (SS) and the natural enamel **[L. 10–16].**

MATERIAL AND METHOD

The five nanocomposite materials that were selected for the study, which demonstrate very good aesthetic properties, are based on innovative technologies and can constitute an alternative to traditional fillings. The tested composites were the following:

- Enamel Plus HRi Function – a material with mechanical properties similar to gold, recommended in raising occlusion;
- Essentia – a composite material with the following structure: enamel structure (nanohybrid with prepolymerized filler), dentin structure (microhybrid with the addition of a nanofiller);
- G-aenial – a nanocomposite with prepolymerised filler;
- G-aenial X FLO – a liquid composite recommended for all classes of cavities, pre-silanized filler; and,
- Charisma Diamond – a nanohybrid universal composite.

Tribological processes in the SS result in contact between the hard tissues of opposing teeth. Natural, physiological wear consists of flattening the cusps and their slopes in the teeth of the maxilla and the mandible under the influence of biomechanical forces. The same processes are subjected to teeth restoration with reconstructive materials.

Samples in the form of ø ¼" discs with a thickness of 1/16" were made, with 15 pieces for each material. The discs were made in standardized ø¼" holes on washers. The methodology of production was compliant with clinical procedures for the reconstruction of the defect. The material preparation process, the application process, and the curing process with the LED lamp (exposure time 20s) were carried out exactly according to manufacturer recommendations. After curing, the samples were pre-made and then polished. After completing the procedures for the final preparation of samples from composite materials, the assessment of their quality was started. Enamel specimens obtained in the form of discs from normal premolars and molars were used as reference materials. Samples and counter-samples were kept for 48 hours in 0.9% NaCl solution at room temperature.

The tests of the coefficient of friction and wear resistance in sliding contact were conducted in the presence of artificial saliva. Saliva is a medium with a high content of enzymes and plays an extremely important role in the lubrication of cooperating occlusion surfaces **[L. 17].** Tribological tests were carried out on a Roxana Machine Works apparatus, using a friction pair consisting of a ball and three discs made from the tested material. The counter-specimens were standard ½ inch diameter ceramic balls made of zirconium oxide with a deviation of 0.00013 mm according to ASTM F2094-02a. The microstructure of the surface layer before the wear process and in the wear defect zone was recorded using a Hitachi S-3400N scanning microscope.

The geometry of the research node approximated the spatial system found in the SS **[L. 18]**. It allowed us to imitate the change in wear intensity observed during *in vivo* studies, which is associated with a change in the contact surface area. Due to the tribological extortions present in the chewing conditions, the following test parameters were adopted:

- Rotational speed = 200 RPM±5 RPM,
- Operation temperature = 36.6°C±1°C;
- Load = 300 N±3 N,
- Duration = 30 min±5 s.

The average wear defect diameter was a measure of anti-wear properties of the tested materials. During the tests, continuous friction torque was recorded, and, on this basis, the coefficient of friction was determined.

The results were statistically analysed using Statistica 13.1 software (StatSoft).

The following were determined:

1) Descriptive statistics (average, median, min, max, std. deviation),
2) Normality of variable distribution (Shapiro-Wilk, Kolmogorov-Smirnov test),
3) Analysis of variance tests (ANOVA),
4) Test of multiple post-hoc comparisons (Tukey, Bonferroni).

The statistical significance level was assumed as $p = 0.05$.

RESULTS

Coefficients of friction of the tested materials are varied (**Fig. 1**).

There are no significant differences between the materials concerning the results of the friction coefficient tests in the initial phase of the procedure (300 s). The coefficient of friction is 0.07–0.12. It remains at the 0.10 level until the end of the run for G-aenial and for Essentia. Slightly higher friction coefficient values of 0.18 were found for G-aenial X FLO. Higher and comparable values were found for Enamel Plus HRi Function and Charisma Diamond. In the final phase, the coefficients of friction stabilized at the level of 0.68 for Enamel and 0.63 for Charisma, respectively. The assessment

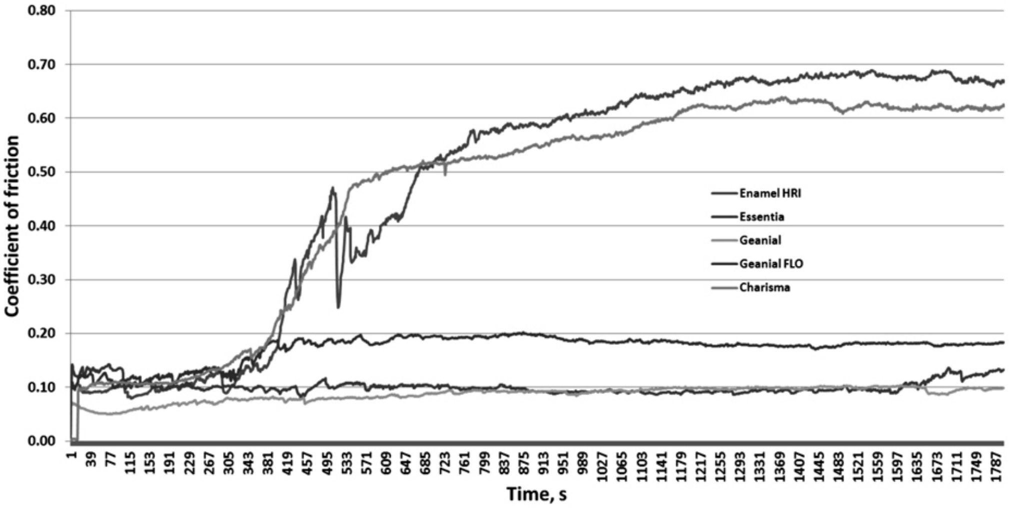

Fig. 1. Representative coefficients of frictions for the tested composites in cooperation with ZrO2
Rys. 1. Reprezentatywne współczynniki tarcia badanych kompozytów we współpracy z ZrO_2

of coefficients of friction leads to the conclusion that materials G-aenial and Essentia are characterized by the least resistance in sliding friction. In the results of the wear resistance tests, the smallest defects were obtained for G-aenial (1.84 mm) and G-aenial X FLO (1.80 mm) (**Fig. 2**). The mean defect value of both materials was very similar and remained within the same range of variation. In the Essentia material, the value of the defect was higher and demonstrated a mean value of 2.14 mm. The highest mean defect values were demonstrated by Charisma Diamond (2.40 mm) and Enamel Plus HRi Function (2.44 mm). The mean defect values of these two materials remained within the same range of variation. One can see that both materials, i.e. Charisma Diamond and Enamel Plus HRi Function, show the weakest tribological properties in terms of the coefficient of friction and wear resistance. The wear resistance of the tested materials was characterized by the following parameters of descriptive statistics: mean value, standard deviation, and result distribution (**Fig. 2, Table 1**). Examples of histograms for Enamel Plus HRi Function and G-aenial are also shown, indicating normal distribution. Insignificant difference between the diameters of the defects was obtained between the materials, Enamel Plus HRi Function and Charisma Diamond, as well as G-aenial and G-aenial X FLO. All other differences in mean defect diameters show statistical significance (**Tab. 2**).

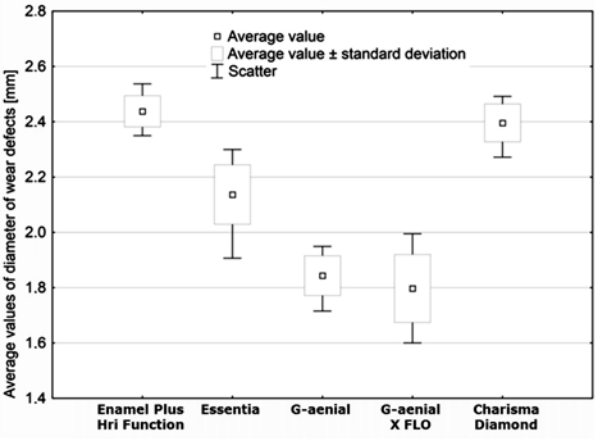

Fig. 2. Tests of wear resistance of tooth restoration materials
Rys. 2. Badania odporności na zużycie materiałów do odbudowy zębów

Table. 1. Comparison of friction coefficient results for the materials tested
Tabela 1. Zestawienie wyników współczynnika tarcia dla badanych materiałów

Tested material	Statistical parameters for description				
	Number of samples	Average value	Minimum value	Maximum value	Standard deviation
Enamel Plus HRi Function	30	2.43	2.35	2.53	0.056
Essentia		2.13	1.90	2.29	0.107
G-aenial		1.84	1.71	1.94	0.072
G-aenial X FLO		1.79	1.60	1.99	0.122
Charisma Diamond		2.39	2.27	2.49	0.068
Enamel		3.14	2.91	3.34	0.13

Table 2. Statistical analysis results of the *post hoc* ANOVA test (HSD Tukey)
Tabela 2. Wyniki analiz statystycznych testu ANOVA post hoc (HSD Tukeya)

Tukey's HSD test Approximate probabilities for post hoc tests Error: MS intergroup =0.00794, df = 145.00					
Material	Enamel Plus HRi Function	Essentia	G-aenial	G-aenial X FLO	Charisma Diamond
Enamel Plus HRi Function		0.000017	0.000017	0.000017	0.364301
Essentia	0.000017		0.000017	0.000017	0.000017
Geanial	0.000017	0.000017		0.253911	0.000017
Geanial X FLO	0.000017	0.000017	0.253911		0.000017
Charisma Diamond	0.364301	0.000017	0.000017	0.000017	

Surface morphology tests were performed for polymer nanocomposites. Selected SEM images of the surface layers developed after the polymerization process (at the clinical level), and after the wear test (within the defect) are presented at the same magnifications (**Figs. 3, 5, 7, 9, 11**). A fine grain structure and scratches resulting from the clinical polishing process are visible in the Enamel Plus HRi material. Scratches deepen within the area of wear-related defect (**Fig. 4**). There are cracks, and numerous sites of scaling are visible in the defect zone, which constitute about 25% of its area (**Fig. 3b**). Perhaps zirconium nanoparticles require modification that will improve the wear parameters of this material.

Essentia material demonstrates a very geometrically varied configuration of particles both within the surface layer after the polymerization process and clinical development and in the wear-related defect (**Figs. 5** and **6**). The edge of the defect and the defect itself are free of scaling and scratches.

SEM images of the G-aenial composite show a pre-polymerized filling with an extremely diverse geometry and with very high percentage share in the composite matrix (**Fig. 7**). The top layer has no scratches at the level of the clinical development. Slight scratches with no visible scaling occur in the area of wear defect. The edge of the wear-related defect is irregular (**Fig. 8**).

At the stage of clinical development and after wear, the G-aenial X FLO composite is characterized by a homogeneous structure, which may result from the presilanized filler used in its structure (**Figs. 9** and **10**). In both areas, nanoparticles and characteristic comparable scratches, no scaling, and a regular defect edge are evident.

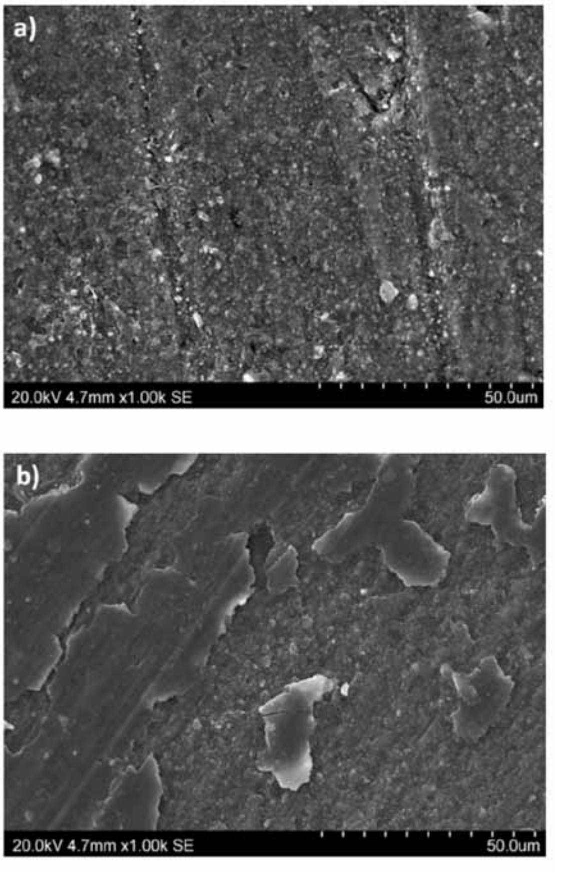

Fig. 3. SEM images of the Enamel Plus HRi Function composite: a) the surface layer developed at the clinical level, b) the surface layer in the defect after the wear test

Rys. 3. Obrazy SEM kompozytu Enamel Plus HRi Function: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w skazie po badaniu zużyciowym

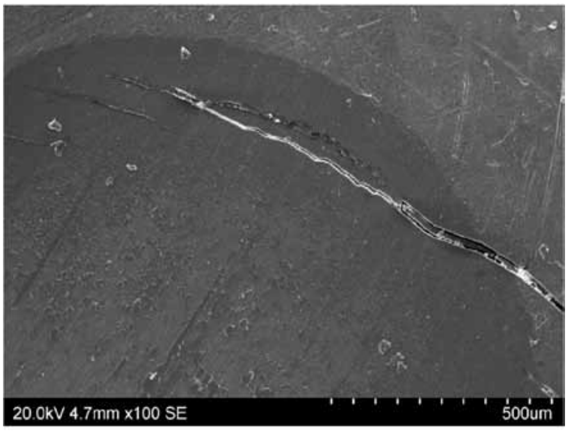

Fig. 4. SEM image of the border zone between the defect and the area before the wear test in the Enamel Plus HRi Function composite

Rys. 4. Obraz SEM strefy granicy między skazą a obszarem przed badaniem zużyciowym w kompozycie Enamel Plus HRi Function

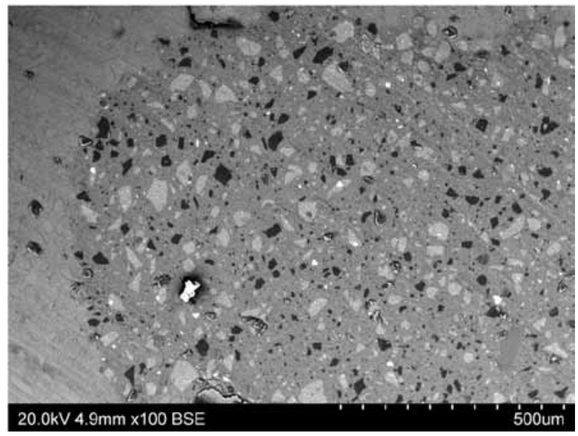

Fig. 6. SEM image of wear defect edge in the Essentia material

Rys. 6. Obraz SEM brzegu skazy zużyciowej w materiale Essentia

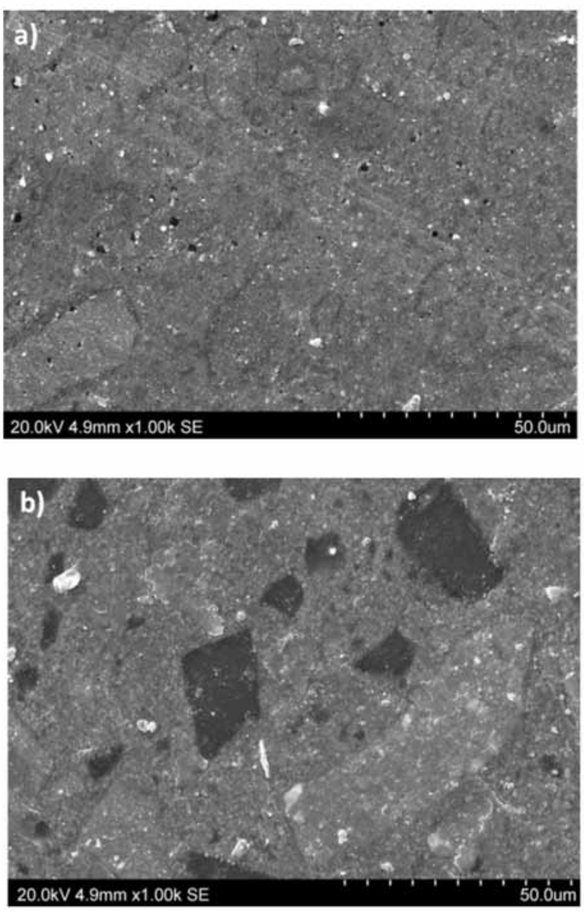

Fig. 5. SEM images of the Essentia composite: a) the surface layer developed at the clinical level, b) the surface layer in the defect after the wear test

Rys. 5. Obrazy SEM kompozytu Essentia: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w skazie po badaniu zużyciowym

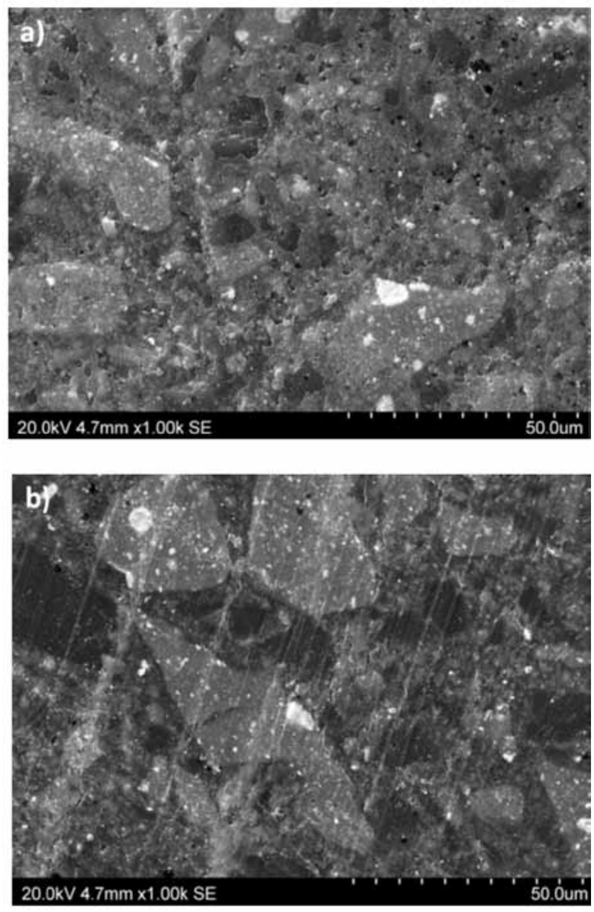

Fig. 7. SEM images of the G-aenial composite: a) the surface layer developed at the clinical level, b) the surface layer in the defect after the wear test

Rys. 7. Obrazy SEM kompozytu G-aenial: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w skazie po badaniu zużyciowym

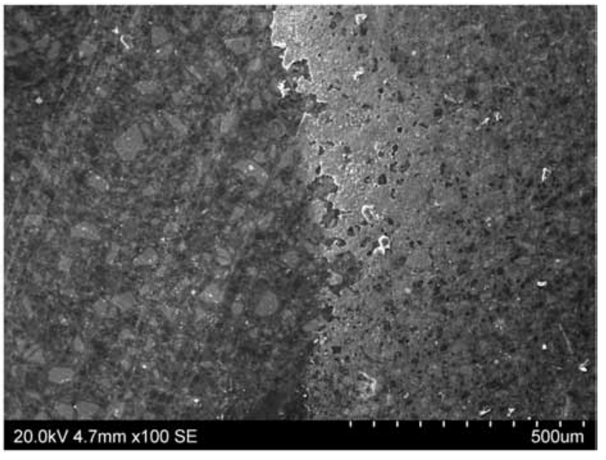

Fig. 8. SEM image of the border zone between the defect and the area before the wear test in the G-aenial composite

Rys. 8. Obraz SEM strefy granicy między skazą a obszarem przed badaniem zużyciowym w kompozycie G-aenial

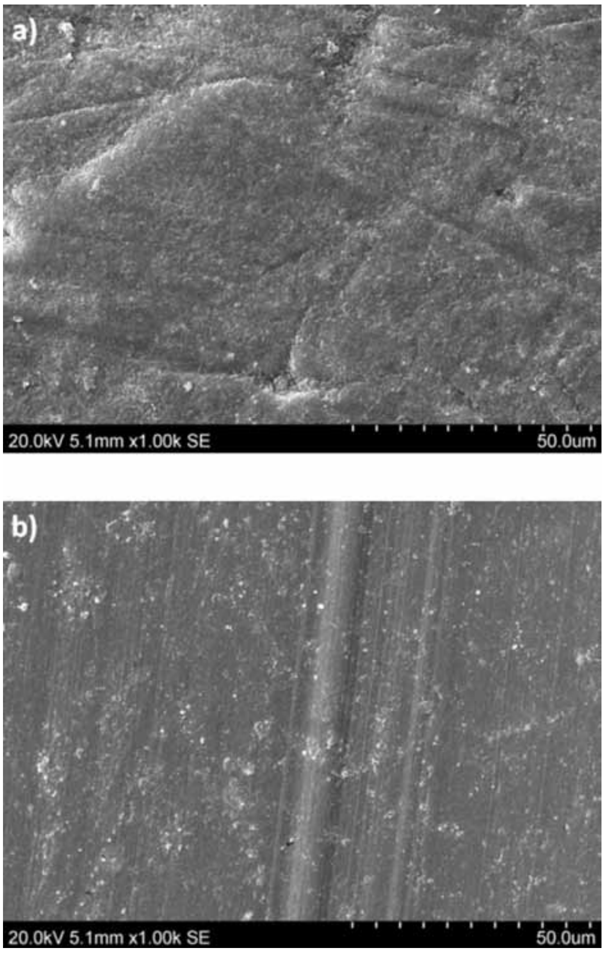

Fig. 9. SEM images of the G-aenial X FLO composite: a) the surface layer developed at the clinical level, b) the surface layer in the defect after the wear test

Rys. 9. Obrazy SEM kompozytu G-aenial X FLO: a) warstwa wierzchnia opracowana na poziomie klinicznym, b) warstwa wierzchnia w skazie po badaniu zużyciowym

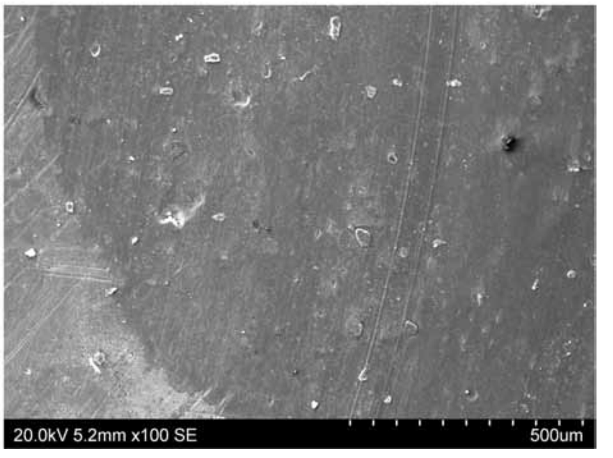

Fig. 10. SEM image of wear defect edge in the G-aenial X FLO material

Rys. 10. Obraz SEM brzegu skazy zużyciowej w G-aenial X FLO

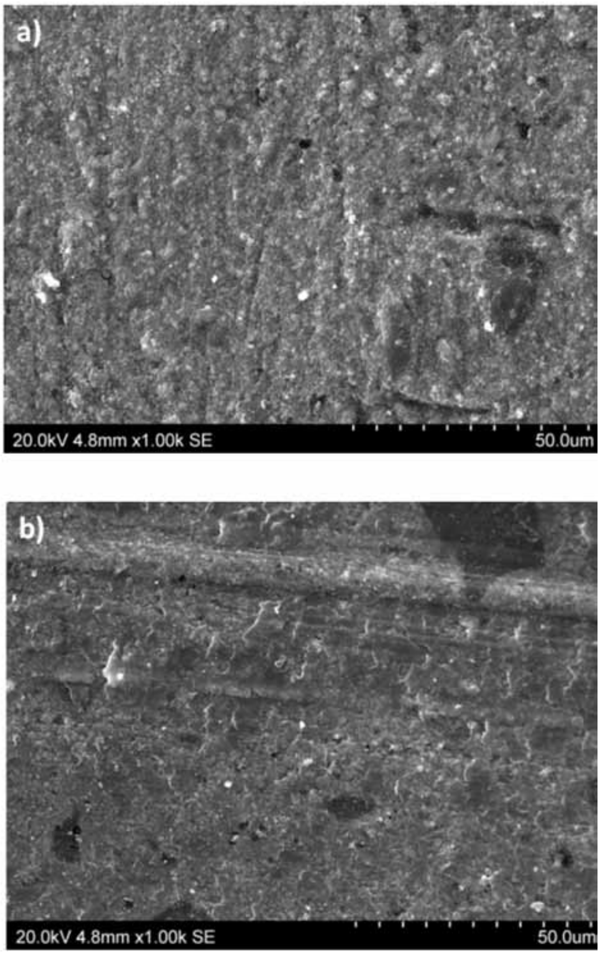

Fig. 11. SEM images of the Charisma Diamond composite: a) the surface layer developed at the clinical level, b) the surface layer in the defect after the wear test

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

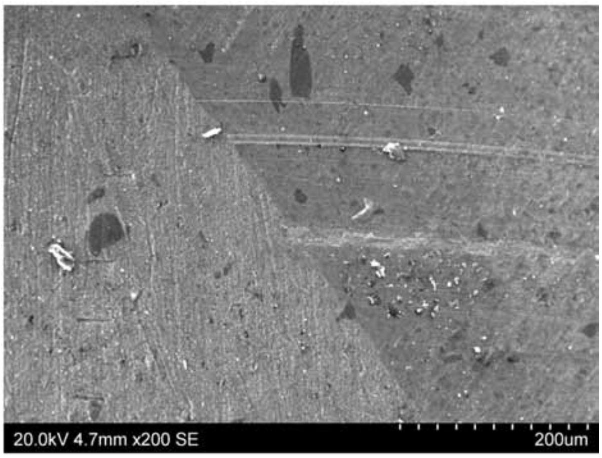

Fig. 12. SEM image of the border zone between the defect and the area before the wear test in the Charisma Diamond composite

Rys. 12. Obraz SEM strefy granicy między skazą a obszarem przed badaniem zużyciowym w kompozycie Charisma Diamond

The most irregular top layer in SEM images was found in the case of Charisma Diamond material (**Fig. 11**). Irregular structure was observed at the stage of clinical development of the surface layer and after the wear process (within the defect). Surface scratches and damage in the form of exfoliation and deep wipes were visible (**Figs. 12** and **13**).

DISCUSSION

Materials for conservative restoration of hard tooth tissues must meet many physical and mechanical requirements. One of them is the smoothness of the surface and gloss comparable to enamel which reduces the deposition of plaque and ensures aesthetic effect. The gloss of the surface layer of the filling involves removing its scratches. The tongue feels a rough surface at 20 μm scratches, and the sensation of smoothness is ensured with 2 μm scratches. The surface smoothness and gloss of the surface layer are required not only in the initial phase of the restoration, but also in the conditions of tribological adjustment wear when using the tooth provided with filling **[L. 6, 19–23]**. This is an important requirement in all classes of cavities, but mostly in the rebuilding of chewing surfaces, because there is a concentration of pressure resulting from occlusion in this zone, and the wear process is the most intense. Numerous *in vitro* tests have demonstrated that the smoothest surface layer is obtained immediately after polymerization of the material before its development **[L. 24, 25]**. In clinical settings, it is often necessary to remove excess material in order to adjust the shape of the filling to the occlusion conditions.

The surface layer structure of resin-based composite materials depends on the size and percentage composition of filler particles. During tribological processes, these particles can be eliminated, which means leaving empty spaces in the structure of the filling. Spaces can affect the increase in the roughness of the surface layer and accelerate wear **[L. 18, 26–29]**. At the same time, polymerization shrinkage is smaller with more suitable filler particles present in the matrix.

A particularly interesting effect was identified in the G-aenial composite by prepolymerizing the filling particles, in the G-aenial X FLO material by presilanizing the filling particles, and in the Essentia material, due to the very large geometric particle differentiation. The introduction of the nanofiller and the stabilization of nanoparticles in the cured structure may result in very good functional parameters. In the foregoing materials, not only the optimal structure was found in SEM images before and after wear, but also very low resistance to movement in sliding friction and low tribological wear. Coefficients of friction of materials ranged from 0.09 to 0.68, and wear defects were in the range of 1.5-2.5 mm. The coefficient of friction of the enamel was at the level of 0.48-0.62, and the resistance to wear measured by the wear defect was at 3.14 mm **[L. 8]**. All composites tested by introducing the nanofiller were characterized by adequate properties for clinical applications in the context of cooperation and enamel adaptation wear.

CONCLUSIONS

The size of the tribological wear depends on the structure of the material obtained in the technological process of making the composite and the surface layer and structure obtained in the clinical restoration process. The process of wear and the transformation of the surface layer that take place during the adjustment processes in the SS are also of essential importance. Materials for restoration should be characterized by the intensity of wear smaller or similar to enamel, the possibility of transferring high pressure in focused contact, and the durability and reproducibility of the correct top layer **[L. 11, 13, 18, 30]**. In chewing conditions, the superficial layers of fillings and opposing teeth are subject to a constant wear process, which should not affect the accelerated wear of the opposed occlusive surface.

The applied test method combining the biomechanical analysis of wear resistance with microstructure analysis enables the assessment of tribological properties in the case of new nanocomposite materials.

All tested materials are suitable for clinical applications in the restoration of the anterior teeth and teeth in the lateral segment of the arch.

The following materials deserve mention in the restoration of cavities: G-aenial, G-aenial X FLO and Essentia, which are characterized by low coefficient of friction values, high resistance to wear, and a lack of scaling and scratches in the wear defect.

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