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Influence of fibre reinforcement on selected mechanical properties of dental composites

AGATA M. NIEWCZAS[1]*, JOLANTA ZAMOŚCIŃSKA[1], ANETA KRZYŻAK[2], DANIEL PIENIAK[3], AGATA WALCZAK[3], GRZEGORZ BARTNIK[4]

[1] Department of Conservative Dentistry with Endodontics, Medical University of Lublin, Poland.
[2] Department of Airframe and Engine, Polish Air Force Academy, Dęblin, Poland.
[3] Department of Applied Mechanics, Main School of Fire Service in Warsaw, Poland.
[4] Department of Mechanical Engineering and Automation, University of Life Science in Lublin, Poland.

Purpose: For splinting or designing adhesive bridges, reconstructive composite structures with increased mechanical properties owing to embedded reinforcement fibres are used. The aim of this article was to determine the influence of glass and aramid fibres on the mechanical strength of composites reinforced with these fibres. *Methods*: Two polymer-ceramic microhybrid materials: Boston and Herculite were tested. Three types of reinforcement fibres were used: aramid (Podwiązka) with a single layer weave, a single layer weave glass fibre (FSO) and triple layer weave glass fibre (FSO evo). Tests were conducted in accordance with the requirements of ISO 4049:2009. The following material types were chosen for research: Boston, Boston + Podwiązka, Herculite, Herculite + Podwiązka, Herculite + FSO and Herculite + FSO evo. The scope of research included: flexural strength σ_B, bending modulus of elasticity ε_B and work to failure of the reinforced composite W_{fb}. Additionally, microscopic observations of fracture occurring in samples were made. *Results*: In comparison: the Herculite (97.7 MPa) type with the Herculite + FSO evo (177.5 MPa) type was characterized by the highest strength. Fibre reinforcement resulted in decreasing the elasticity modulus: Herculite + reinforcement (6.86 GPa; 6.33 GPa; 6.11 GPa) in comparison with the Herculite (9.84 GPa) and respectively Boston + reinforcement (10.08 GPa) as compared with the Boston (11.81 GPa). *Conclusions*: Using glass fibres increases flexural strength of the test composites. Using aramid fibres does not change their strength. The elasticity modulus of the reinforced reconstructive structures decreases after application of either type of fibres. However, their resistance to the crack initiation increases.

Key words: *mechanical properties, ceramic-polymer light cured composites, fibre reinforcement*

1. Introduction

Traditional dental ceramic-polymer composites are characterized by low abrasiveness and an elasticity modulus similar to modulus of dentin [1]. It was proven by tests that these composites are sensitive to crack initiation and propagation. These cracks are initiated particularly in the area of tensile stress concentration and lead to a composite failure. Therefore, in dental treatment, for instance, in the case of splinting or adhesive bridges, additional reinforcements in the form of twisted fibres are frequently introduced (Fig. 1). Currently, reinforcement ribbons made of fibres are widely used. Although experience gained during clinical trials indicates the limited sustainability of these applications, using reinforcement fibres is justified for therapeutic and economic reasons [2]–[5].

Reinforcing materials differ in terms of type, properties, shape and functions. Carbon, glass, aramid reinforcements in roving form connected to each other without twist into fabrics of variable weave are used [6].

Glass fibres are distinguished by high tensile strength with a relatively low flexural modulus [7]. The strength of a single glass fibre depends on its diameter. A larger diameter can be the reason for the reduction of strength [8]. Glass fibres have good wettability in the case of implementing so-called preparation, which

* Corresponding author: Agata M. Niewczas, Department of Conservative Dentistry with Endodontics, Medical University of Lublin, ul. Karmelicka 7, 20-081 Lublin, Poland. Tel: 48607169068, e-mail: agatan117@wp.pl

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increases surface tension (this enables a good connection with a composite). The adverse property of glass fibres is high sensitivity to water. In dental procedures, glass fibres are used in splinting loosen teeth, especially in the front part of the dental arch. They constitute a good reinforcement for the reconstruction of teeth crowns. They are useful wherever a greater strength of reconstructed part of tooth tissue is required. Moreover, inserts of glass fibre are used as additional reinforcement with standard composite material in rebuilding teeth after the endodontic treatment. Thus the risk of root breaking is effectively reduced.

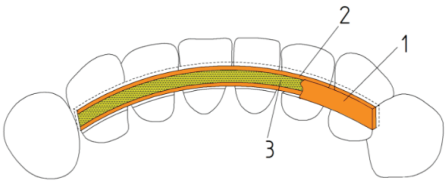

Fig. 1. Scheme of dental arch splinting:
1 – composite layer covering the splint,
2 – the first layer of composite covering teeth,
3 – strand of fibres embedded in the composite structure

Aramid fibres are characterized by low density, good mechanical properties and high resistance to corrosion. Their disadvantage is low bending strength. The study showed an increase in strength of the aramid fibre reinforced composites when the reinforcement was located on the tensile side of the sample in the shape of a beam. Additionally, there was an increase in the elasticity modulus when the reinforcement was located on the side of compression [8]. In other studies, it was found that this type of composite has the highest strength when fibres are placed in one direction, on the tensile and compression sides [9]. The placement of fibres where maximum stress occurs is the best solution as for as mechanical strength is concerned. Unfortunately, such an order of fibres does not correspond to the clinical situation, in which the fibre is constantly placed between layers of the resin based composite (Fig. 1). Studies concerning an impact of the direction of fibres show that composites in which fibres were placed in one direction have a higher strength than those in which the fibres were in both directions (weaves) [10].

Reviewing the available literature dealing with the conditions of laboratory tests the strength of dental composites, it can be concluded that standards conditions in the oral cavity for reconstructing have not been fully developed yet. In dentistry, simply transferring methods applied in technical mechanics is not justified. One of the most difficult problems is the impact of the samples' scale. In the case of such small applications as used in dentistry dimensions, direction of fibres and position of placing reinforcement may be crucial. The way of preparing samples has also a big impact on test results. Therefore, such studies should be carried out on samples prepared by experienced dentists. Moreover, the way of their preparation should be similar to the clinical one.

The objective of the present study was to determine the impact of glass and aramid fibres on the mechanical strength of reconstructive dental structures made of selected composite materials.

In cooperation with the manufacturer (LFS Arkona) in the field of innovative solutions for reinforcing dental composites with fibres, the authors studied the selected strain-stress characteristics dependent on reinforcement architecture and structure on the example of three new reinforcement fibres applied combined to well-known universal polymer-ceramic composites.

2. Materials and test method

Research was conducted on two chosen commercial dispersive composites reinforced with molecular fillers (Fig. 2): Boston (LFS Arkona) and Herculite (Kerr). These materials are the base for producing ribbon reinforced composite structures in combination with three different types of reinforcing fibres. Basic data concerning the test composites are presented in Table 1.

Table 1. Composites used in research

Material	Boston	Herculite
Manufacturer	LFK Arkona	Kerr
Type	Microhybrid composite	Microhybrid composite
Type of filler particles	Baria-alumina-silica glass, igneous silica, titanium dioxide	Inorganic mineral fillers with average particle size of 0.6 microns
Weight of filler particles	78%	79%

Boston composite is based on a mixture of bisphenol A diglycidyl ether dimethacrylate, diurethane dimethacrylate and triethylene glycol dimethacrylate with particulate of solids of baria-alumina-silica glass, igneous silica and titanium dioxide. Herculite is based on a matrix with methacrylate esters with titanium dioxide and zinc oxide. Boston composite has a flex-

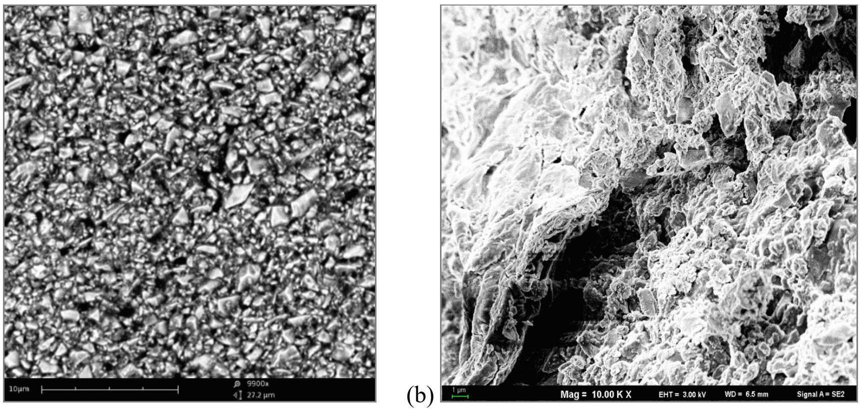

Fig. 2. SEM images of microstructure of dental composites tested:
(a) microhybrid composite Herculite XRV, (b) composite with microfiller Boston

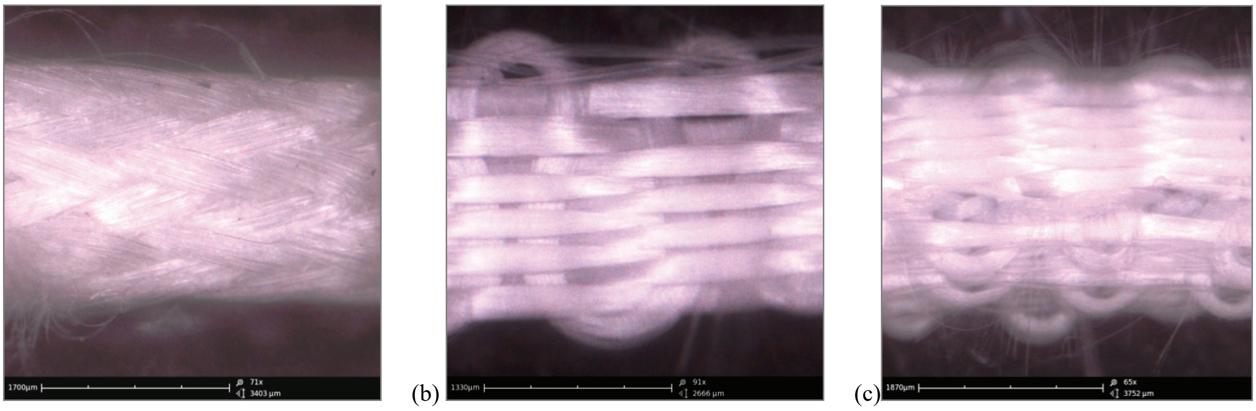

Fig. 3. Fibre architecture: (a) a Braid weave type of aramid fibres, Podwiązka, (b) a single-layer weave of fibre glass Fibre Splint Ortho, (c) triple-layer weave of fibre glass Fibre Splint Ortho evo

ural strength of approximately 140 MPa, while the flexural strength of Herculite composite is 150 MPa. In the following research, these commercial composites were reinforced with aramid fibres with a single layer weave with a trade name Podwiązka (LFS Arkona), as well as glass fibres with a single Fibre-Splint Ortho (FSO) and triple layer weave glass fiber Fibre-Splint Ortho Evolution (Polydentia) FSO evo. A preparation of methacrylate resin was applied on aramid fibres, which enables for better bonding with the ceramic-polymer composite. Photographs in Fig. 3 show the architecture of weave of the test fibres. The polymerization process was implemented by irradiation with a diode lamp for 40 s.

Research described in this paper has been carried out in accordance with the requirements of ISO 4049:2000 – technical support [11], [12]. The study was based on a strength test under the conditions of a three-point bending [13], [14]. According to the above standard, the support span was 20 mm in length. This corresponds to a clinical situation where tooth arch segment is subjected to reinforcement including 3–4 teeth, or filling the teeth loss of two neighbouring teeth with adhesive bridge [15]. Test samples were made in the form of rectangular prism beams with dimensions 2 mm × 2 mm × 25 mm. Tests were conducted at a crosshead speed of 0.5 mm/min. Radius of supports and stem implementing extortion changes to the position of beam were 1 mm. The list of test samples is presented in Table 2.

Table 2. List of the test materials

Item	Number of samples	Material type
1	10	Boston
2	10	Boston reinforced with Podwiązka fibre
3	10	Herculite
4	10	Herculite reinforced with Podwiązka fibre
5	10	Herculite reinforced with FSO fibre
6	10	Herculite reinforced with FSO evo fibre

Bending stress (σ_B) was calculated based on of the following formula

$$\sigma_B = \frac{3PL}{2bd^2} \quad \text{[MPa]}$$

where:

P – load during the trial [N],

L – support span [mm],

b – sample width [mm],

d – sample thickness [mm].

Bending modulus of elasticity (E_B) was calculated with the following equation

$$E_B = \frac{mL^3}{4bd^3} \quad \text{[MPa]}$$

where:

m – slope of the initial straight-line portion of the load-deflection curve,

L – support span [mm],

b – sample width [mm],

d – sample thickness [mm].

Bending work (W) was defined as follows

$$W = \int_0^y P dy \quad \text{[Nmm]}$$

where:

P – load during the trial [N],

y – beam deflection [mm].

Test results were statistically analysed by means of Statistica 10.0 calculation software. Descriptive statistics and the value of standard deviation were determined by assuming 0.95 level of confidence.

3. Results

The test conducted include the following mechanical properties of materials: σ_B – flexural strength, E_B – elasticity modulus at bending, W_{fB} – work to failure of the reinforced composite. Test results are presented in Figs. 4 through 7.

Mean values of flexural strength of composites not reinforced with fibres ranged from 80 MPa to 98 MPa. On the other hand, mean values of flexural strength of composites reinforced with fibres ranged from 79 MPa to 178 MPa. However, it should be noted that the strength of composites reinforced with the Braid (Podwiązka) fibre was similar to that of non-reinforced composites (Fig. 4). Herculite composite reinforced with FSO evo fibre displayed the highest flexural strength and scatter of results.

Non-reinforced composites were characterized by the greatest value of the bending modulus of elasticity (9.84÷11.81 GPa). Only the Boston composite reinforced with Podwiązka fibre displayed a similar modulus value. The bending modulus of elasticity for composites reinforced with FSO and FSO evo fibres was lower by half and fell within range from 6.11 to 6.86 GPa (Fig. 5).

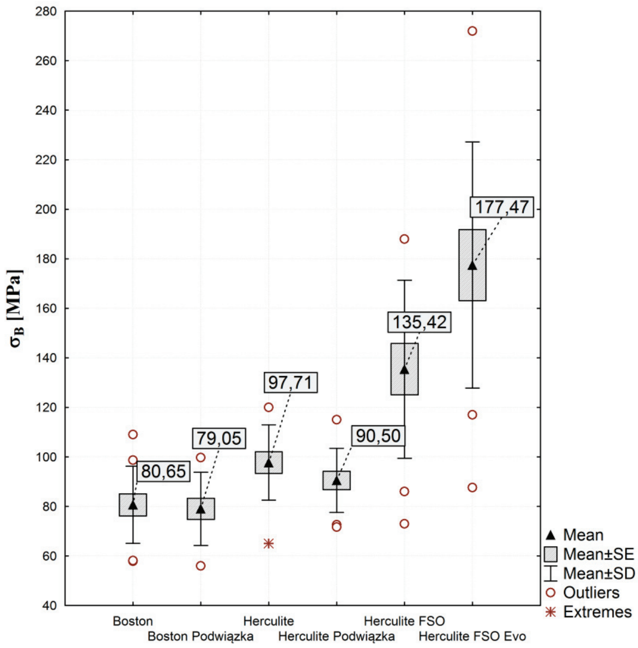

Fig. 4. Box plot comparing values of flexural strength of composites reinforced with fibres

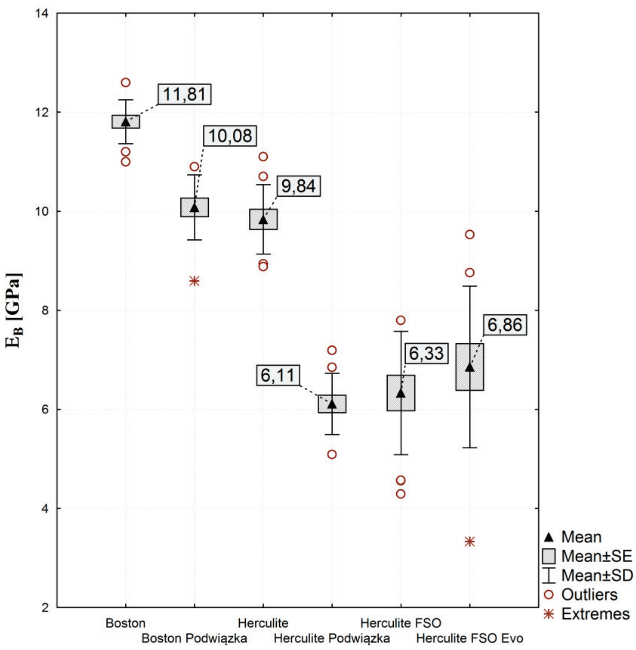

Fig. 5. Box plot of bending modulus of elasticity of composites reinforced with fibres

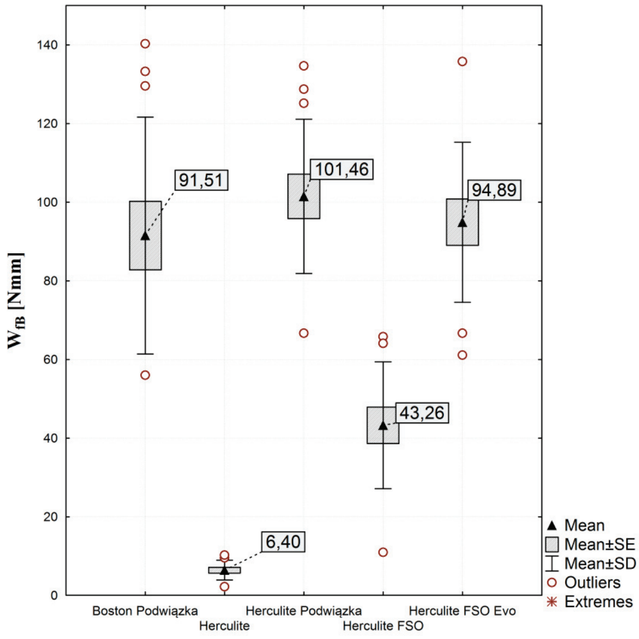

Fig. 6. Box plot comparing work required to destroy reinforced composite

The lowest mean value of the work to failure was obtained for the non-reinforced material Herculite (Fig. 6). The highest mean values were obtained for the composite reinforced with Podwiązka fibre and Herculite reinforced with FSO evo fibre.

Figure 7 shows the load-deflection curve during the flexural strength test. The course of the presented characteristics indicates the brittle nature of failure only in the case of non-fibre reinforced composites. The process of strain and deformation of reinforced materials proceeds in stages. In the case of the Boston composite reinforced with Podwiązka fibre in the first stage until reaching the threshold of strength, there is no noticeable irresilient deformation. Damage appears suddenly with an accompanying decrease in force till approximately 30% F_{max}. Then, the process slows down, strengthens and there is an increase in force to around 50% F_{max}. A similar process of failure can be observed in the case of Herculite composite reinforced with Podwiązka fibre. However, the decrease in force in the first stage is lower and there are cyclic increases in the force value during the deformation stage. The strain and deformation process of Herculite composite reinforced with FSO fibre occurs at low values of deformation. Irresilient deformation is small and thus the course of the process is similar to the characteristics of brittle materials. There is a difference in the process of the strain and deformation in the case of Herculite composite reinforced with the multi-layer FSO evo weave. Structures with this system demonstrate lower elasticity modulus. The irresilient part of the characteristic is visible. Additionally, slight changes in force, which do not influence the further increase in strength are noticeable. After reaching the F_{max} value, which is the biggest among the test materials, there is a slow decrease in force with a significant maintenance of the strength level of the final stage.

Figure 8 presents microscopic images of fractures in test samples of composites under three-point bending conditions.

Figure 8a presents fracture in a beam made up from Herculite composite reinforced with FSO fibre. The dominant impact of tensile stress is noticeable. Figure 8b presents the fracture of a beam made of Herculite composite reinforced with FSO evo fibre. Relatively minor beam damage in the tensioned area can be observed. Figure 8c shows the crack of the top layer in the beam made of Boston composite reinforced with Podwiązka fibre. Figure 8d presents the de-adhesion of Podwiązka fibre from Herculite composite. Figure 8e presents surface cracks of

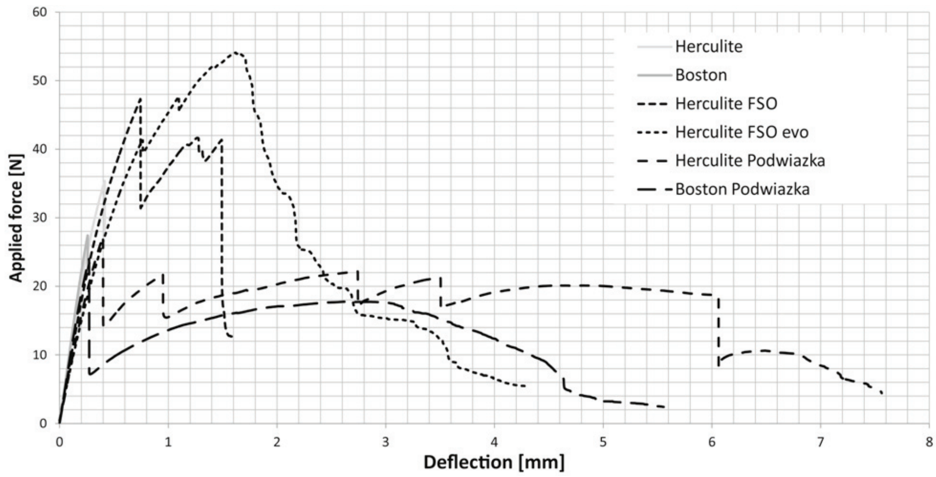

Fig. 7. The load (in [N]) as a function of deflection (in [mm])

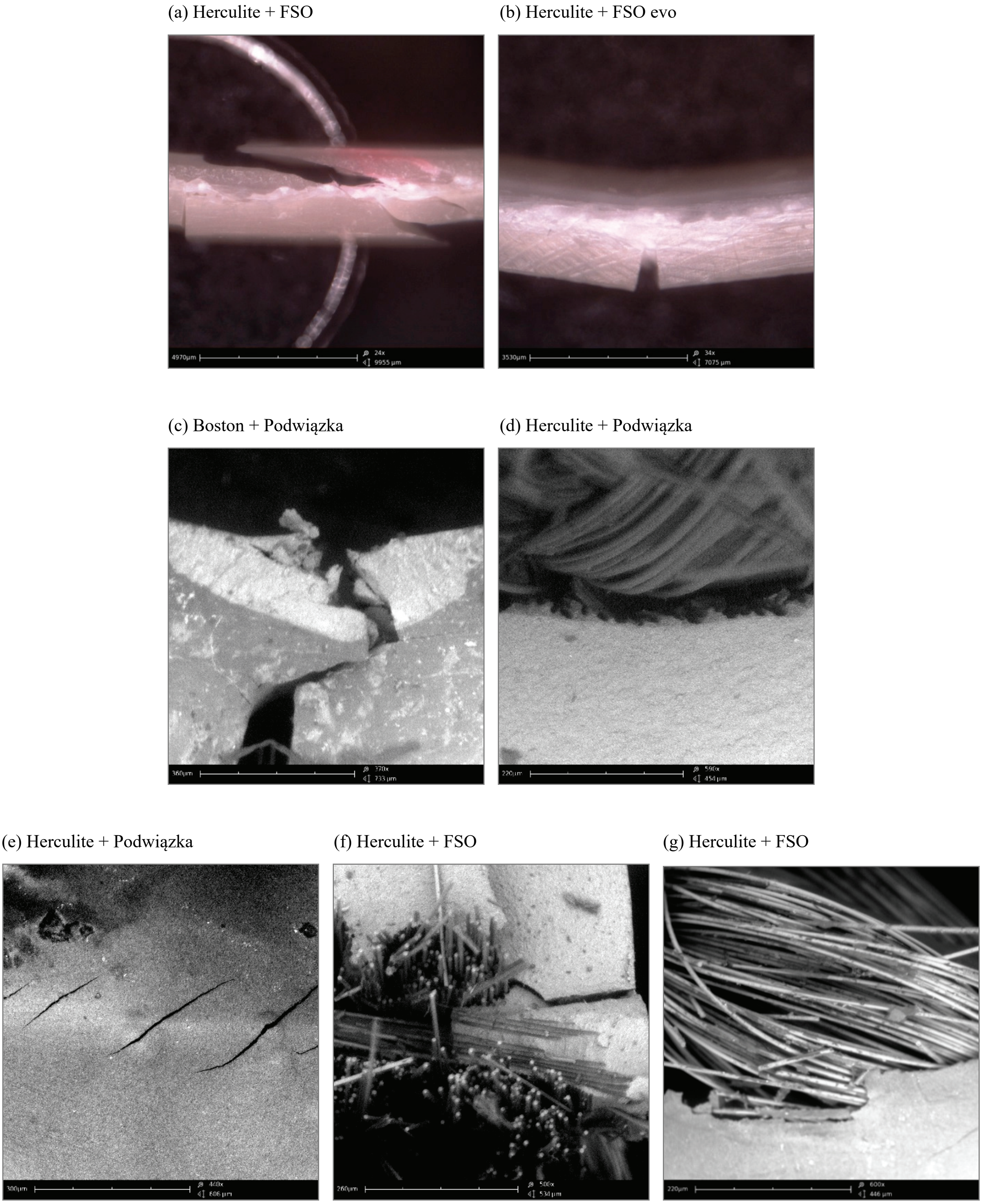

Fig. 8. Microscopic images of fractures in composites

Herculite composite reinforced with Podwiązka fibre. Figure 8f presents the fracture of the beam made of Herculite composite reinforced with FSO fiber. Figure 8g presents the damage of the beam made of Herculite material reinforced with FSO fibres. The damage is the de-adhesion of fibres being in direct contact with the composite. Torn fibre weave is visible.

4. Discussion

Interpreting test results in terms of flexural strength of materials, it can be claimed that the impact of fibre reinforcement varies. After Podwiązka fibres were used, both composites tested: Herculite and Boston, showed a similar level of strength as non-reinforced composites. Nevertheless, embedding FSO glass fibres, especially in the triple layer weave resulted in a substantial increase in strength (Fig. 4).

It can also be concluded that using reinforcement in the following order of layers: composite–fibre weave–composite leads to decrease of elasticity modulus of the composite element. A lower bending modulus of elasticity decreases the susceptibility to crack initiation. In the majority of cases under study, it is essential to use more energy to create and develop a crack, and subsequently, to destroy the connection between the dispersive composite and fibres. Similar results are also published by other researchers [17]–[19]. In the case of application in the dental arch, lower stiffness may limit the susceptibility to crack initiation. However, it is unlikely to be of importance for the subsequent stage of damage, which is delamination between the fibre reinforcement. In order to ensure high stiffness and coherence of the application, it is most beneficial to use the composite reinforced with aramid fibres with Braid (Podwiązka) weave (Fig. 5). In the case of applying aramid fibres, interlaminar damage and defects in the form of lack of continuity of the dispersive infiltrating of composite in the space between fibres directly contribute to limiting of endurance of the reconstruction in dentistry. Based on the obtained test results, it can be concluded that glass fibres with a multilayered FSO evo weave are characterized by universality, which results in good mechanical properties of the layer system (fibre-composite). Greater susceptibility of the multilayered FSO evo structure limits the development of primary structural faults. Among other things, it results from a relief in tension that is, decreasing the tension gradient between layers.

Analyzing the course of failure of the test materials, subsequent stages following each other can be distinguished. In the first subcritical phase small regular cracks of the beam in the tensioned part were observed in the composite reinforced with multilayered FSO evo weave (Fig. 8b). In the case of the remaining test materials, cracks occurred at the bottom of the layer of the beam that was exposed to tension, caused transferring the load to a higher degree by reinforcing fibres (Fig. 8b). In the case of Boston composite, sometimes the upper part of the dispersive composite was damaged, which was caused by the local gradient of stress in this layer of the beam. This phenomenon is likely to result from a number of factors: good connection of dispersive composite and fibres as well as considerable inconsistency in the stiffness of Podwiązka fibre weave made of dispersive composite (Fig. 8c).

Independently of the reinforcement architecture in the initial stage of the critical phase in many cases, (examples in Fig. 8d, f, g), a complete loosening between the composite and the fibres is occurred. Empty spaces between the fibres, that result from insufficient infiltration of the dispersive composite are visible. These are large structural defects. The presence of such defects results from high viscosity of the dispersive composite which prevents its movement especially in weaves with tightly placed fibres, for instance, Podwiązka. What in this case cannot be observed is a border layer, which in polymer composites is responsible for even transfer of external load to reinforcement fibres. Progressive failure of the composite and reinforcement (Fig. 8d) is sometimes preceded by microcracks emerging at the border of layers (Fig. 8e), it refers to the composites reinforced with Podwiązka and FSO eve fibres to a lower extent. In many samples reinforced with FSO evo weave, the lack of delamination may result from multilayered reinforcement weave. The lack of cracks may be the results of mutual moving of the dispersive composite layers. However, in the case of Podwiązka fibres stiff reinforcement was most likely achieved by proper preparation of the surface of fibres which consisted in applying a layer of high viscosity methacrylane resins. Catastrophic failure at maximum force occurred only in the case of implementing glass fibre reinforcement, most frequently FSO. It proceeded simultaneously with the decomposition of the weave structure (Fig. 8g) and sometimes tearing of fibres (Fig. 8f) or extensive damage and separation of composite material into several parts (Fig. 8a) were observed. Failure shown in Fig. 8a can be caused by the aforementioned faults located between reinforcement fibres. These faults emerge at the time of applying and connecting materials by the dentist.

5. Conclusions

1. Applying glass or aramid fibres influences mechanical properties of reinforced composite structures in a diverse way. Tension and deformation

characteristics are mainly dependent on the properties of reinforcing fibres and not of the dispersive composite.

2. The greatest strength was obtained while applying reinforcement made of FSO evo glass fibres. It is a structure charecterized by average stiffness and the biggest tolerance to internal damage.
3. Braid weave made of aramid fibres does not provide good overall strength. However, it increases the bending modulus of elasticity. The process of complete failure is slow and occurs during considerable deformation. A tight, single layer weave prevents the penetration of the composite with filler into spaces between fibres.
4. It should be added that further research should involve tests of reinforcement made of aramid fibres with multilayered and multidirectional weave since changing the fibre architecture gives the greatest potential for shaping mechanical properties of the material. Moreover, the scope of further research should be extended to cyclic mechanical and thermal loads simulating conditions existing in the oral cavity.

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