Mechanical strength assessment of aramid, glass and aramid-glass hybrid fibers reinforced dental photopolymer

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Abstract: Strength parameters of dental photopolymer reinforced with long glass and aramid as well as aramid-glass hybrid fibers was to comparison. Static strength tests of 40 light-curing composite specimens were performed with the use of Zwick 1435 testing machine and testXpert V.8.1 software. Flexural strength of aramid fiber reinforced polymer increased nearly three times, whereas flexural strength of polymer reinforced with glass fiber – twice.

Keywords: mechanical strength, polymer, glass fibers, aramid fibers, aramid-glass hybrid.

Ocena wytrzymałości mechanicznej światłoutwardzalnego polimeru stomatologicznego wzmocnionego włóknami aramidowymi, szklanymi oraz hybrydowymi aramidowo-szklanymi

Streszczenie: Porównano właściwości wytrzymałościowe światłoutwardzalnego polimeru stomatologicznego wzmocnionego długimi włóknami szklanymi, aramidowymi lub hybrydowymi aramidowo-szklanymi. Statyczne próby wytrzymałościowe 40 próbek kompozytu na zginanie przeprowadzono przy użyciu maszyny Zwick 1435 z wykorzystaniem programu testXpert V.8.1. Wytrzymałość na zginanie polimeru wzmocnionego włóknami aramidowymi zwiększyła się prawie trzykrotnie, a w wypadku wzmocnienia włóknami szklanymi – dwukrotnie.

Słowa kluczowe: wytrzymałość mechaniczna, polimer, włókna szklane, włókna aramidowe, włókna hybrydowe aramidowo-szklane.

For many years, photopolymer resins have been widely used in many areas of dentistry. The most commonly used composites are easily applicable, produce the desired aesthetic effect, exhibit lower polymerization shrinkage and strong adhesion to enamel and dentin. However, due to their relatively low flexural strength of approximately 90 MPa, attempts have been made to increase composite strength with a wide variety of reinforcing materials [1–4]. High tolerance to mechanical damage is particularly significant in composite restorations placed in premolar and molar region of jaws, where occlusal forces reach the value of 270 N [5]. There are many methods of improving mechanical strength properties of composite materials. Studies on fiber reinforced composites (FRCs) are promising. They are becoming more widely used as an alternative to conventional fixed prosthetic restorations in specific clinical cases [3, 4].

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The aim of the study was to compare changes in chosen strength parameters (maximum bending force, strain for maximum bending force, the bending strength and Young's modulus) of dental composite depending on reinforcing it with long glass, aramid fibers as well as aramid-glass hybrid.

EXPERIMENTAL PART

Materials

A2 shade Gradia Direct Posterior light-cured composite resin (GC Corporation, Tokyo, Japan) – [7,7,9 (or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate, ytterbium trifluoride, (octahydro-4,7-methano-1H-indenediyl)bis(methylene) bismethacrylate] and G Bond self-etching light-cured adhesive (GC Corporation, Tokyo, Japan) – [2-hydroxylethyl methacrylate, urethanedimethacrylate and catalysts] were used in the study.

The specimens of composite were reinforced with long glass and aramid. The weight ratio of the particular fibers in aramid-glass hybrid was 1 : 1. All fibers were in the

T a b l e 1. Comparison of composition and parameters of the glass and aramide fibers used

Fiber	Glass fiber	Aramid fiber
Manufacturer	ATG (France)	Kevlar DuPont (USA)
Composition, wt %	59 SiO_2; 12.1–13.2 Al_2O_3; 22–23 CaO; 3.1–3.4 MgO; 0.6–0.9 Na_2O; 0.5 other	Poly(*p*-phenylene terephthalamide)
Basis weight, g/m^2	200	200
Diameter of elementary fiber, μm	16	15
Roving linear mass, tex	200	200
Poisson number	0.22	0.36

form of roving, which meant that the fiber hanks were interconnected without twists. The composition and parameters of selected fibers are presented in Table 1.

Sample preparation

The samples were prepared in accordance with the procedure for making the fibre reinforced composite (FRC) bridges in a dental laboratory. The process of specimen preparation consisted of 3 steps:

Fibers cleaning

25 mm long, 2 mm wide and 0.2 mm thick fiber bundles were cleaned with acetone (Alchem, Poland). The aim of the acetone wash was to remove potential contaminants from fiber surface created in the manufacturing process. After completing the cleaning procedure, fibers were dried for 2 hours at a temperature of 50 °C.

Fibers coating with adhesive

After drying, fibers were placed on a glass plate and impregnated with adhesive – G Bond. The weight ratio one fiber bundle/G Bond was 1 : 3. In order to protect them from light, all the specimens were covered with aluminum foil. After 5 minutes visible light-induced polymerization of fiber bundles was performed for 40 seconds with the use of Woodpecker LED B curing light (Guilin Woodpecker Medical Instrument Co. Ltd., China) – light intensity (irradiance) > 1000 mW/cm^2 in the wavelength range of 400–480 nm.

Preparation of composite specimens

In order to maintain comparable specimen sizes, silicon mold of established shape was created in accordance with the standard PN-EN ISO 4049:2010. Every specimen was cuboid in shape and was 25 ± 0.1 mm long, 2 ± 0.1 mm wide and 2 ± 0.1 mm thick. Fiber reinforced specimens were fabricated using hand lay-up method. The first 0.5 mm layer of Gradia Direct Posterior light-cured composite resin placed in the silicone mold. Then the pre-impregnated fibers were laid. In the end, a second layer of the same composite resin was placed to fill the mold (Fig. 1). In addition, rolling was used to remove air bubbles and facilitate penetration of the resin.

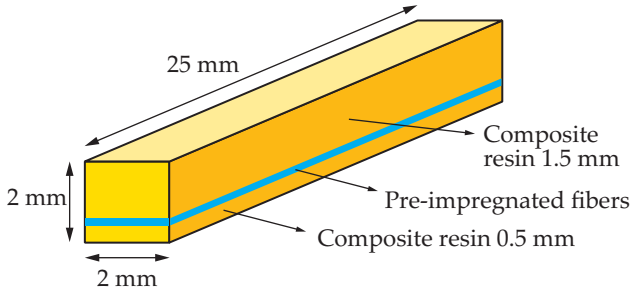

Fig. 1. Shape and measurement of specimens with laminar composite structure

The study involved a total of 40 specimens (including 10 controls). The study group included 3 series of 10 composite specimens reinforced with synthetic fiber (comprising 2% of the specimen weight) arranged in configurations described in the aim of the study.

The control group consisted of a series of 10 unreinforced composite specimens.

Each specimen was subsequently bilaterally polymerized for 2 minutes with the Woodpecker LED B curing light. After hardening, specimens were removed from the mold. Wet grinding with no. 1200 FEPA (14 μm) (Struers, Ballerup, Danmark) removed excess material.

Specimens from each group were stored dry at room temperature for 24 hours before testing.

Methods of testing

Static three-point bending test was performed using the same devices and in the same way as in our previous study [6]. Bending speed of 1 mm/min was constant while the distance between supports were 20 mm apart.

The use of the testXpert V.8.1 software (Zwick/Roell GmbH & Co. KG Germany) allowed the determination of four basic strength parameters: maximum bending force (F_{max}), strain for maximum bending force (εF_{max}), limit stress determining the bending strength (σ) and Young's modulus (E) – constant elasticity characterizing stiffness of the tested material.

The statistical analysis was performed using the R software package, version 3.4.4 (The R Foundation for Statistical Computing, GNU GPL).

RESULTS AND DISCUSSION

Average values of the measured mechanical parameters were calculated along with estimation of

T a b l e 2. Results of Shapiro-Wilk normality test

Index	Samples	Shapiro-Wilk		
		Statistics	*df**)	*p*
F_{max}	Control group	0.93	10	0.56
	Glass fibers	0.90	10	0.20
	Aramid fibers	**0.84**	**10**	**< 0.05**
	Aramid-glass hybrid	0.97	10	0.90
ε F_{max}	Control group	0.98	10	0.95
	Glass fibers	0.91	10	0.26
	Aramid fibers	**0.60**	**10**	**< 0.01**
	Aramid-glass hybrid	0.95	10	0.64
σ	Control group	0.93	10	0.55
	Glass fibers	0.90	10	0.20
	Aramid fibers	**0.82**	**10**	**< 0.05**
	Aramid-glass hybrid	0.97	10	0.90
E	Control group	0.97	10	0.89
	Glass fibers	0.96	10	0.76
	Aramid fibers	0.90	10	0.20
	Aramid-glass hybrid	0.97	10	0.85

*) *df* – degrees of freedom, *p* – probability value.

T a b l e 3. Descriptives statistics for analylized variables

Index	Samples	*M*	*SD*	Skewness	Curtosis
F_{max}, N	Control group	21.19	3.28	-1.46	2.64
	Glass fibers	47.12	13.98	-0.89	-0.07
	Aramid fibers	61.58	12.98	-1.60	4.46
	Aramid-glass hybrid	53.73	9.31	0.42	-0.28
ε F_{max}, mm	Control group	0.65	0.09	0.32	-0.64
	Glass fibers	1.30	0.41	-0.63	-0.66
	Aramid fibers	1.89	0.51	-2.73	8.11
	Aramid-glass hybrid	2.08	0.29	0.36	-0.04
σ, MPa	Control group	79.48	12.29	-1.46	2.65
	Glass fibers	176.70	52.43	-0.89	-0.07
	Aramid fibers	231.83	48.39	-1.69	4.87
	Aramid-glass hybrid	201.47	34.89	0.42	-0.28
E, GPa	Control group	4.96	0.62	0.05	-0.87
	Glass fibers	7.15	1.33	-0.48	-0.40
	Aramid fibers	7.55	1.00	-0.80	-0.37
	Aramid-glass hybrid	6.98	0.80	-0.64	0.21

M – medium, *SD* – standard deviation.

the combined standard uncertainty for the coverage factor k = 2. Thus, the probability that the result of any measured value was within the range of $\bar{x} \pm 2 \cdot S_x$ (S_x – standard deviation, $\bar{x}$ – arithmetic average) amounted to 0.9545. Thus, the lue of the coverage factor k was equal to standardized variable, read from the tables of normal distribution in natural sciences, corresponding to the confidence level α = 0.95 [7, 8].

In the first order, the congruence between analyzed variables distribution and theoretical normal distribution was checked with a Shapiro-Wilk test. The results indicated that in the case of 3 indexes: F_{max}, ε F_{max} and σ specimens reinforced with aramid fibers characterized with distribution significantly violating the assumption of normal distribution. In the case of Young's modulus glass group was violating this assumption (Table 2).

Additionally, descriptive statistics were analyzed especially in respect of skewness and kurtosis, bearing in mind rule of thumb saying that values falling out from <-1;1> interval signalize violating normal assumption (Table 3). Those results were generally congruent with normality tests. According to the fact, that only exceptional variables, with regard to each index, were signalizing significant departure from normal distribution the parametrical ANOVA test was used in order to verify the hypothesis about the differences between specimens. In order to control family-wise error Bonferroni correction was used. Cohen's d statistic was used to measure effect size.

A comparative assessment of test results showed aramid fiber reinforced composite to have the highest flexural strength [medium (M) = 61.58, standard deviation (SD) = 12.98, $p < 0.05$] which corresponded to the maximum values of yield stress within the limits of linear elasticity (M = 7.55, SD = 1.00). Intermediate results were obtained with aramid-glass hybrid composite (M = 53.73, SD = 9.31, $p > 0.05$). All significant differences characterized with Cohen's higher than 0.8 indicating strong effect sizes.

The results of ANOVA indicated that there were significant differences between analyzed specimens in all indexes which were set under scrutiny. The strongest differences (according to effect size measure η^2 p) were observed in case of ε F_{max} [distribution statistics (F) = 29.74, probability (p) < 0.001, partial etha square (η^2 p) = 0.75 and the smallest ones, but still with high level of significance, were observed in case of Young's modulus – F = 5.487, $p < 0.001$, η^2 p = 0.35. Control group (M = 21.19, SD = 3.28) characterizes with the lowest level of F_{max}, which was significantly lower than all other specimens.

The highest value of σ was demonstrated for samples reinforced with aramid fibers (M = 231.83, SD = 48.39, $p < 0.05$). Intermediate results were obtained with aramid-glass hybrid composite (M = 201.47, SD = 34.89, $p > 0.05$) in comparison to samples reinforced with glass fibers (M = 176.70, SD = 52.43, $p > 0.05$). The highest deflection values that corresponded to the lowest values of Young's modulus in relation to the control test were demonstrated by samples reinforced with aramid-glass hybrid.

Material properties ensuring mechanical strength parameters that allow its reasonable and clinical use are determined by many factors. Mechanical strength of a material is not determined by the sum or mean value of individual mechanical properties of its constituents, *i.e.*, its matrix and fiber. These include, in particular, the type and amount of organic matrix bonding material and geometric characteristics of the fiber such as its length, diameter and distribution.

FRCs are the materials of low resting weight. They are also characterized by high long-term static and dynamic compressive strength values regardless of strains distribution in the stomatognathic system during cyclic mastication [2, 9, 10]. A great number of researchers have proven parallel alignment of fibers composed of multiple constituents to strengthen the composite material [2–5, 9, 10]. Numerous studies have shown carbon, polyethylene, glass or aramid fibers to provide even an eightfold increase in flexural strength of the composite with 8–12 wt % contents of fibers in the specimens [11–16]. In this study, we demonstrated that dental polymer strengthening with 2 wt % of aramid fibers increased its bending strength by 191%. Aramid fibers owe their high strength mainly to the type of weave used in the process of fabrication.

The use of fiber of any type requires special attention to be paid to fiber careful filtration with the use monomers or specific adhesive materials. Proper fiber preparation ensures its good adhesion to composite [2, 3, 9, 12, 17–19]. Location and distribution of fibers in a specimen is particularly important, and has been confirmed by numerous authors [10, 14–16, 20, 21]. During specimen bending, part of the material directly exposed to an applied load undergoes compression, whereas the opposite side – stretching. A complex composite has better strength under compression compared to stretching, therefore cracking occurs first and it is followed by fracture at the stretching site [14, 21, 22]. When designing fiber-reinforced prosthetic restorations in which fibers run in a parallel fashion, it is recommended that one of the fiber bundles run opposite to the applied force, *i.e.* usually on the mucosal side. Such fiber pattern is recommended by authors of the study, as well as by other researchers [10, 14–16, 23].

Studying the reinforcement effect of glass fibers in composites Lassila *et al.* observed a lower degree of their polymerization when compared with unreinforced materials during the same light-curing time [24, 25]. Therefore, preparation of the study group specimens involved longer polymerization time than that recommended by manufacturer for the complete hardening of specimen and polymerization of resin filling the spaces between fiber bundles.

A great number of variables determining mechanical properties of the studied material may generate different results depending on a research center conducting similar studies. As observed by Karbhari *et al.* the differences may emerge even if the same type of material is used [26].

CONCLUSIONS

Within the limitation of this *in vitro* study, it concludes that the flexural strength of the aramid fiber reinforced dental photopolymer increased nearly three times whereas flexural strength of polymer reinforced with glass fiber – twice. Further studies are needed to evaluate whether aramid-glass fiber hybrid is an effective modality in improving the properties of dental composite significantly.

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