STUDY OF CERAMIC-POLYMER COMPOSITES RELIABILITY BASED ON THE BENDING STRENGTH TEST

OCENA NIEZAWODNOŚCI KOMPOZYTÓW CERAMICZNO-POLIMEROWYCH NA PODSTAWIE PRÓBY WYTRZYMAŁOŚCI NA ZGINANIE

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Abstract: In this paper the reliability assessment of structural reliability of the selected light-cured dental composites based on the biaxial flexural strength test results has been presented. A two-parameter Weibull distribution was applied as a reliability model in order to estimate probability of strength maintenance in the analysed population. Weibull distribution parameters were interpreted as a characteristic material strength (scale parameter) and structural reliability parameter in terms of ability to maintain strength by each of specimen from the general population (shape parameter). 20 composite specimens underwent strength tests, including 2 "flow" type composites and 2 standard composites (with typical filler content). "Flow" type composites were characterized with lower characteristic strength and higher structural reliability comparing to other studied composites.

Keywords: dental composites, strength, reliability analysis

Streszczenie: W artykule przedstawiono ocenę niezawodności strukturalnej wybranych światłoutwardzalnych kompozytów stomatologicznych na podstawie wyników testu wytrzymałości na zginanie dwuosiowe. Wykorzystano dwuparametrowy rozkład Weibull'a jako model niezawodności w celu oszacowania prawdopodobieństwa zachowania wytrzymałości materiału w analizowanej populacji. Parametry rozkładu Weibull'a interpretowano jako wytrzymałość charakterystyczną materiału (parametr skali) oraz wskaźnik niezawodności strukturalnej w sensie zdolności do zachowania charakterystycznej wytrzymałości przez każdy z egzemplarzy populacji generalnej (parametr kształtu). Próbie wytrzymałościowej poddano po 20 próbek kompozytów, w tym 2 kompozyty typu „flow” oraz 2 kompozyty standardowe (z typową ilością wypełniacza). Kompozyty typu „flow” charakteryzowały się niższą wytrzymałością charakterystyczną oraz wyższą niezawodnością strukturalną w porównaniu z pozostałymi badanymi kompozytami.

Słowa kluczowe: kompozyty stomatologiczne, wytrzymałość, analiza niezawodności

1. Introduction

Clinical studies indicates that one of the main reasons of dental composite fillings failures is composite cracking [2, 12, 18]. Therefore, in *in vitro* studies conducted in order to determine characteristics of the studied material in the conditions close to reality, an important aspect is a mechanical strength. It depends on many factors such as microstructure and composite failure process, as well as type of research method and environment [6, 15]. According to ISO 4049 standard [14] related to dental composite materials used for re-construction of hard teeth tissues, material strength is determined in three-point flexural strength test. In the literature examples of the use of some other research methods can be found, including biaxial flexural strength test [11, 19, 17].

Biaxial flexural strength study is included in technical standard ISO 6872 [13]. In this method a tensile stress occurs in central part of the specimen. It decreases by dispersion over larger area, thus by increasing radius between area where stress occurred and centre of the sample [1, 5]. The advantage of this study method is a round shape of the specimen. Occurrence of the maximum stress in the central part of the specimen prevents formation of cracks on the edges. What is more, the area of stress occurrence is smaller than in case of three-point bending test, therefore there is a lower probability of stress occurrence on the surface with defects. It is worth mentioning that polymerization process of the round samples is more repeatable. Additionally, a direction and distribution of internal stresses generated as a result of this process and polymerization shrinkage has a lower influence on the result of strength test.

Mechanical strength describes material's behaviour subject to static loads. One of the most important factors influencing ceramic-polymer composites strength is size and distribution of random deviations in the structure of the single specimen of the studied material. In order to perform a general assessment of the material it is necessary, besides strength determination in the studied random sample, to determine probability of strength behaviour in the studied population. For the forecast of the above defined structural reliability Weibull distribution can be applied [3, 9, 10, 15]. Weibull distribution modulus (m , shape parameter) is taken as a distribution of material strength in the considered manufactured series [7]. High value of shape parameter indicates probability of lower number of random defects occurrence in the composite and potentially higher reliability in the clinical use. Weibull distribution parameter σ_0 , (scale parameter), is described as a characteristic material strength value, which reflects 63.2% cases of material failure.

The objective of this paper was to perform the structural reliability evaluation of the selected light-cured ceramic-polymer composites based on the biaxial flexural strength test.

2. Material and method

The following four light-cured ceramic-polymer composites were studied: Filtek Z500, Filtek Flow, Flow Art and F2. Basic data of the above mentioned materials are included in tab. 1.

Tab. 1. List of studied materials

No	Material	Manufacturer	Type	Type and size of filler particles	Content of filler particles (% wt.)	Content of filler particles (% vol.)
1	Filtek Z500	3M ESPE (USA)	Nanocomposite	SiO ₂ 20nm, ZrO ₂ /SiO ₂ 5-20nm, (0.6-1.4µm clusters)	78,5%	59,5%
2	Filtek Flow	3M ESPE (USA)	Microhybrid "flow" type	ZrO ₂ /SiO ₂ 0.01-6µm average: 1.5µm	68%	47%
3	Flow Art	Arkona L.F.S. (Poland)	Microhybrid "flow" type	barium-alumino-silicate glass, fumed silica, titanium dioxide average: 0.76µm	64%	No data
4	F2	Arkona L.F.S. (Poland)	Microhybrid	fluoro-barium-alumino-silicate glass average: 0.90 µm	79%	No data

Biaxial flexural strength test was conducted on 20 composite specimens of each type ($N=20$). The specimens were disc-shape with diameter of 13 mm and thickness of 2 mm, they were exposed to LED lamp for 40 sec. Before tests were initiated the specimens had been kept in artificial saliva at 37°C for 3 months. Tests were conducted using strength machine Zwick/Roell Z100. The specimens were placed on three steel balls (fig. 1). Supports were located on the circle perimeter with circle diameter of 10 mm, angle between the supports was 120°. The specimens were loaded through the flat piston (fig. 1). Crosshead speed was 0.5 mm/min.

Fig. 1 Schematic diagram of the piston-on-three-ball biaxial strength test

Based on the biaxial bending test a maximum tensile strength was determined σ_{BI} , using the following formula:

$$\sigma_{BI} = \frac{-0.2387 P(X-Y)}{h^2} \quad (1)$$

$$X = (1 + \nu) \ln \left(\frac{r_2}{r_3} \right)^2 + \left[\frac{1-\nu}{2} \right] \left(\frac{r_2}{r_3} \right)^2 \quad (2)$$

$$Y = (1 + \nu) \left[1 + \ln \left(\frac{r_1}{r_3} \right)^2 \right] + (1 - \nu) \left(\frac{r_1}{r_3} \right)^2 \quad (3)$$

where:

P - failure load, N,

h - specimen height, mm,

ν - Poison's ratio, for all composites $\nu = 0,24$ [12],

r_1 - radius of the support ball circle, $r_1 = 5$ mm,

r_2 - radius of loaded area, $r_2 = 0,6$ mm,

r_3 - specimen's radius, mm.

3. Study results

Descriptive statistics of study results

Table 2 presents descriptive statistics and Weibull distribution parameters determined based on strength study results: specimens number (N), average value (average), median (Q2), quartile one (Q1) and three (Q3), dispersion, standard deviation and coefficient of variation, as well as Weibull modulus (m) and characteristic strength (σ_0). For the analysis purpose strength results were grouped based on the composite type: Filtek Z500, Filtek Flow, Flow Art, F2 (respectively FZ500, FFlow, FlowArt, F2).

Tab. 2. Descriptive strength statistics and Weibull distribution parameters

Material	N	Average	Q2	Q1	Q2	Dispersion	St.dev	Coeff. Var.	σ_0	m
		[MPa]						[%]	[MPa]	
FZ500	20	116.75	115.31	112.28	126.06	34.47	9.97	8.54	121.24	13.58
FFlow	20	93.10	94.10	91.57	94.83	18.81	4.60	4.94	95.30	22.96
Flow Art	20	85.63	86.69	80.74	88.62	13.87	4.31	5.03	87.683	21.88
F2	20	114.55	114.60	94.47	136.79	80.78	23.33	20.37	123.93	5.79

Reliability analysis

Conducted analysis shall be seen as an attempt to assess the probability of maintaining standards of material strength in the whole population, thus as a repeatability of the obtained results in the whole population.

Reliability analysis was conducted assuming Weibull distribution as a probability distribution model of the failure of studied composites at the biaxial bending test conditions. Failure probability P_f , is described here by Weibull distribution function with the following formula:

$$P_f = 1 - \exp \left[- N \left(\frac{\sigma - \sigma_u}{\sigma_0} \right)^m \right] \quad (4)$$

where:

e - constant ($e = 2.71828\dots$),

N - number of samples,

σ - failure stress,

σ_u - location parameter (stress value corresponding to probability of failure close to 0),

σ_0 - scale parameter,

m - shape parameter.

Number of specimens of each type of material was 20, therefore in the equation (4) it was possible to make a simplification of neglecting N value [4, 16]. Due to a random nature of defects occurrence in the studied composite specimens it was assumed that location parameter $\sigma_u = 0$. After considering the above assumptions the equation (4) will have the following form:

$$P_f = 1 - \exp \left[- \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (5)$$

The above equation after taking the logarithm can be simplified to the linear form:

$$\ln \left[\ln \left(\frac{1}{1 - P_f} \right) \right] = m \ln(\sigma) - m \ln(\sigma_0) \quad (6)$$

where:

$$P_f = \frac{x - 0.5}{N} \quad (7)$$

x - specimen number after "ranking" according to strength values.

Based on the equation (6) a survival probability (P_s) can be obtained, since $1 - P_f = P_s$, thus:

$$\ln \left[\ln \left(\frac{1}{P_s} \right) \right] = m \ln(\sigma) - m \ln(\sigma_0) \quad (8)$$

Equation (8) enables determination of non-parametric survival probability curve P_s , necessary for estimation of Weibull distribution parameters. Intersection point of Y axis by approximation function depends on $-m \ln(\sigma_0)$, while slope parameter of approximation function is a shape parameter of Weibull distribution m [8]. The linear approximation of the strength test results, regression equations and determination coefficients after logarithmic transformation are presented in figure 2.

Fig.2 Approximation of survival probability distribution

Empirical failure function curves (Weibull distribution function) based on strength results of the studied composites is presented in figure 3. In the figure a red dotted line indicates probability level of failure $P_f=0,632$ corresponding to characteristic strength σ_0 .

Fig. 3 Failure function of the bending strength of studied composites

4. Conclusions

- Ceramic-polymer composites have a complex internal structure and usually have some defects (flaws). The defects initiate development of the failures. Application durability is limited by developing failures (resistance of material with higher initial unreliability decreases faster), therefore it is directly connected with internal structure of the composite and load history.
- Descriptive statistics determined based on the results indicate lower strength of "flow" type composites comparing to other studied composites. Similarly, scale parameter of Weibull distribution is lower for "flow" type composites. It is related to lower filler particles content in those materials.
- No significant difference in strength between material with nanoparticles (FZ500) and microhybrid composite (F2) has been observed. Most probably it is related to similarity of materials structures, where the clusters are present in the micro scale of nanoparticles.
- Higher Weibull modulus value of "flow" type composites indicates its probable higher reliability, in the population characterized with lower structure entropy. Additionally, it can be expected that the number and sizes of structural defects in "flow" type composites are lower than in case of other studied materials.
- The results of studies and conducted analysis confirmed necessity of extending strength studies with reliability analysis in order to gain generic data on the characteristics of the studied composites.

5. Literature

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