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TEMPORARY STABILITY OF COMPRESSIVE STRENGTH OF FLOW AND UNIVERSAL TYPE LC PMCCS MATERIALS

ABSTRACT

This paper reports the results of compressive strength and elasticity studies of light-cured polymer matrix ceramic composites (LC PMCCs). The main purpose was to obtain new data on experimental composites and compare them with commercial composites from the world's leading manufacturer. The objective was to investigate the relationship between the content of reinforcing components in the composites studied and the stability of their strength in time, expressed as the number of fatigue thermal cycles.

Keywords: *light-cured polymer matrix ceramic composites (LC PMCCs), compressive strength, linear regression*

INTRODUCTION

Composites are complex materials containing at least two chemically different phases which are visibly separated at the microscopic level, but integrated at the macroscopic level. A composite produced by the integration of phases has different properties than its individual constituents. Composites are commonly reinforced with dispersed particles. One example of dispersion-reinforced materials are light-cured polymer matrix ceramic composites (LC PMCCs). LC PMCCs have a multiphase structure, and are, most often, composed of two basic phases and a photoinitiator. The basic phases are a polymer (organic phase), constituting approx. 20–40% of the material's volume, and ceramic fillers (non-organic phase) dispersed in the first phase, making up approx. 60% (up to 70–80 wt%) of the material's volume. The organic phase is usually based on light-cured methacrylate resins, such as bisphenol A

glycidyl methacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA), and polycarbonate dimethacrylate (PCDMA) [1].

The mechanical properties of LC PMCCs depend on many factors such as their microstructure, damage mechanisms, and the environment in which they are used [2-3]. In the context of medical applications, the mechanical properties of LC PMCCs are crucial. They are related to the material's strength and define what work can be performed on the material [4]. In the case of LC PMCCs used as dental fillings, the occlusive active forces acting in physiological conditions, when sufficiently strong, may result in the material losing its cohesion, and thus lead to its breaking, cracking, crushing, etc. [5]. In other words, the mechanical properties of a composite material are responsible for safe transfer of loads.

Because the investigated composites are used as reconstructive materials in dentistry, their compressive strength is crucial. Studies of similar materials under compression conditions have already been carried out by Ivanisevic et al. [6], who observed that the compressive strength of the LC PMCCs they tested varied within the range between 200 and 400 MPa.

The strength of dental fillings made of LC PMCCs changes in time and depends on their load history, including thermal loads. Dental fillings made of composites with a polymer matrix are subject to thermal cyclic loads in physiological processes, thus their fatigue resistance is an important design factor [7].

Thermal fatigue is defined as gradual degradation of the mechanical properties of a material due to the initiation and propagation of structural defects and cracks caused by changes in internal energy under the influence of a periodically alternating temperature field. Thermal fatigue can occur, among others, in materials characterized by an inhomogeneous structure, with limited freedom of deformation by kinematic ties, as is the case with dental fillings loaded with an additional constant force field or additionally periodically loaded with an alternating force field. The state of stress depends on the effect of mechanical factors, but not solely. The structure of dental fillings is subject to stresses and thermal deformations, which are characterized by a gradient related to the penetration of the thermal front into the centre of the structure. For example, during heating or cooling, changes occur faster on the material's surface than in its centre – the core. Thermal loads originate from the oral cavity environment. Alternating contact with cold and warm liquids and solids can have an influence on the properties of the materials studied.

A typical range of temperatures on the tooth surface in the oral cavity is between 1 and 50 °C [8-9]. However, often teeth are subjected to higher temperatures. Depending on the dental filling studied, some authors report that approx. 6000 thermal loads occur during five years of normal operation [10], others quote the number of 2000 thermal loads sustained over 200 days [11]. Due to the lack of detailed studies, the available literature does not provide full and unequivocal information on the number of cyclic loads in the expected operating time of a dental restoration, which, according to Niewczas et al. [12] is four to eight years. The objective of the present study was to determine the impact of cyclic thermal loading on the residual compression strength of dental fillings.

MATERIALS AND EXPERIMENTAL PROCEDURES

The following LC PMCCs were used in the study: flow type Filtek Flow (FF) and flow type experimental material (FTEM), as well as universal Filtek Z500 (Z500) and universal type experimental material (UTEM). Basic information on the composites studied is given in Table 1.

Table 1. *Ceramic-polymer materials applied in the studies*

Material	Type	Resin	Filler	Filler content % wt.
Z500	Nanocomposite	Bis-GMA, UDMA, TEGDMA, Bis-EMA	SiO_2 particles (20nm) ZrO_2/SiO_2 (clusters 0.6–1.4 μm, particles 5–20 nm)	78.5
UTEM	Microhybrid	Bis-GMA, TEGDMA, Bis-EMA	Bar-aluminum-fluoro-boro-silica glass particles, fire silica and titanium oxide (average size of 0.90 μm)	78
FF	Flow type microhybrid	Bis-GMA, TEGDMA, Bis-EMA	ZrO_2/SiO_2 (0.01–6 μm) particles	68
FTEM	Flow type microhybrid	Bis-GMA, UDMA, TEGDMA, Bis-EMA	Bar-aluminum-fluoro-boro-silica glass particles, fire silica and titanium oxide (average size of 0.76 μm)	64

Bis-GMA – bisphenol A glycidyl methacrylate; UDMA – urethane dimethacrylate; TEGDMA – triethylene glycol dimethacrylate; Bis-EMA– ethoxylated bisphenol A dimethacrylate

Compressive strength tests were performed using a Zwick/Roell Z100 strength testing machine (Fig. 1). The test speed was 10 mm/min and the initial force was 2 N. The test pieces were cylinders with a diameter of 6 mm and a height of 3 mm (Fig. 1).

Compression strength σ_{cmax} was determined as the ratio of load at failure to cross-sectional area of the specimen:

$$\sigma_{cmax} = \frac{P}{A} \; [MPa] \tag{1}$$

where:
P – load [N],
A –cross-sectional area of specimen [mm^2], , d – diameter of specimen [mm].

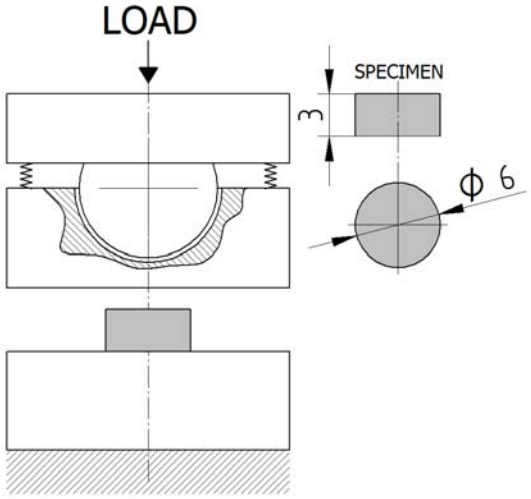

Fig. 1. Compressive strength test

Accelerated tests simulating cyclic thermal loads were performed on a dedicated device. The thermal shocks simulator consisted of two systems: a control and a hydraulic system. The hydraulic system included (Fig. 2) an ultra-thermostat equipped with a heating system, and another ultra-thermostat with a cooling system. Peristaltic pumps were used for pumping a working liquid in and out of the vessel containing the specimens [13-14].

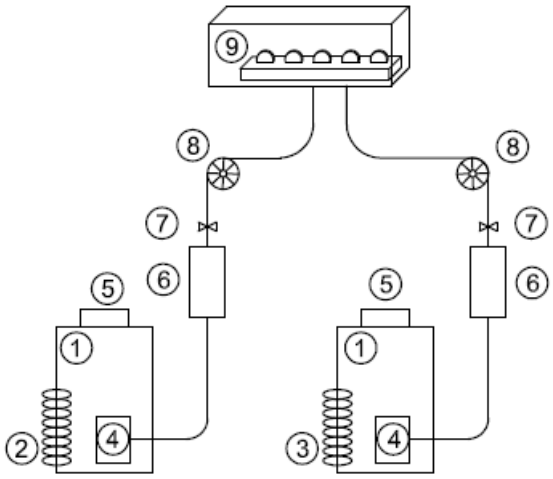

Fig. 2. Schematic of the hydraulic system of the thermal shocks simulator: 1– working liquid tank, 2 – heating system, 3 – cooling system, 4 – rotary pump, 5 – thermostat, 6 – container, 7- shut-off valve, 8 – peristaltic pump, 9 – specimen-holding vessel

During the accelerated tests, the specimens were placed in a special vessel, in which the working liquid with a specified temperature was pumped in and out at specified times. 10 000 thermal cycles were performed, each lasting 201 sec. The times of execution of each stage of the thermal cycle are given in the diagram in Fig. 3. The experiments were conducted in a temperature range of 10 to 70 ^{o}C. Specimen exposure time in liquid with a set temperature was 30 sec. Samples subjected to thermal fatigue were marked with the symbol 10kTC.

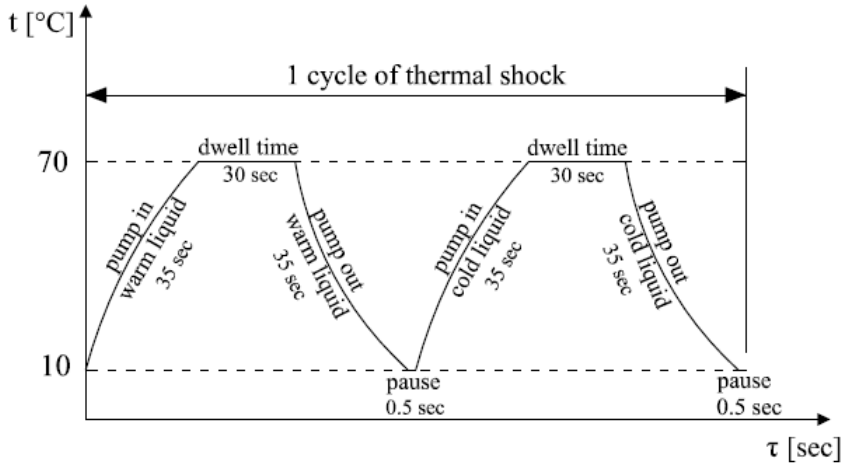

Fig. 3. Operating cycle of the thermal shock simulator

The influence of filler content on selected properties of the composite was calculated using the following general linear regression equation:

$$Y = \beta_0 + \beta_1 X + \varepsilon \,, \tag{2}$$

where:

Y – property studied,

X – filling component in % wt,

β_0, β_1 – structural model parameters,

ε – random variable with normal distribution $N(0, \sigma^2)$ representing external disturbances in the model.

The impact of filler content in the composite on compressive strength σ_{cmax} and Young's modulus E was analysed using linear models (1). It was assumed that the filler content in the composite was between 60 to 80% by weight, i.e. $X \in [60,80]$.The structural parameters of the linear regression models (1) for FTEM, FF, UTEM, and Z500 specimens, as well as for FTEM_10kTC, FF_10kTC, UTEM_10kTC, and Z500_10kTC specimens were determined using the standard least squares method. For each model describing the investigated features, determination coefficients R^2 were found. The significance of the multiple correlation coefficient was investigated using the Fischer–Snedecor test for the linear models (1) describing the relationship between the feature studied and filler content X (the null hypothesis that the multiple correlation coefficient insignificantly differs from zero $H_0: R = 0$, was compared to the alternative hypothesis $H_1: R \neq 0$). The significance of structural parameters was also studied using Student's t-test; the null hypothesis $H_0: \beta_i = 0$ was tested against the alternative hypothesis. The significance level was set at $\alpha = 0.05$.

RESULTS AND ANALYSIS

The results of the compressive strength studies are presented in Table 2, and the results of Young's modulus are given in Table 3. The following parameters are included: mean values, standard deviations, minimum and maximum values, and first and third quartiles. Specimens subjected to thermal fatigue tests are marked with the symbol 10kTC.

Statistical significance was determined by Mann-Whitney U and Kruskal-Wallis tests. The significance level was set at $\alpha = 0.05$. In Tables 2 and 3, two identical capital letters in bold indicate that the difference between the mean values studied was statistically significant. Two identical lower case letters indicate a lack of statistical difference between the mean values studied.

Table 2. *Descriptive statistics of compressive strength*

	Mean	SD	Min	Max	1 Q	3 Q
Z500	423.00$^{\mathbf{Ab}}$	54.26	329.00	509.00	383.50	466.00
Z50010kTC	413.00^{b}	58.79	284.00	479.00	392.00	452.25
UTEM	394.88$^{\mathbf{Aa}}$	48.30	311.00	468.00	359.00	426.00
UTEM10kTC	405.42^{a}	31.00	346.00	461.00	387.50	423.00
FF	351.71$^{\mathbf{CD}}$	68.84	171.00	423.00	324.00	408.00
FF10kTC	320.26$^{\mathbf{D}}$	31.49	245.00	346.00	314.50	340.00
FTEM	277.81$^{\mathbf{Cc}}$	29.23	219.00	312.00	263.25	301.50
FTEM10kTC	284.35^{c}	31.28	196.00	322.00	267.00	307.00

Compressive strength values were higher for the universal type composites than for the flow type materials. The difference between the mean values of compressive strength of UTEM and Z500 was considerable. The difference between mean compressive strengths of the FF and FTEM composites was also noticeable. A highly statistically significant influence of cyclic thermal loads was only demonstrated for Filtek Flow.

Table 3. *Descriptive statistics of Young's modulus*

	Mean	SD	Min	Max	1 Q	3 Q
Z500	2915.26^{Ab}	145.08	2570.00	3130.00	2830.00	3030.00
Z50010kTC	2805.00^{b}	242.39	1980.00	3060.00	2767.50	2935.00
UTEM	2693.53^{Aa}	125.15	2410.00	2890.00	2650.00	2780.00
UTEM10kTC	2620.53^{a}	212.20	2120.00	2850.00	2565.00	2780.00
FF	1925.29^{BC}	288.38	1340.00	2310.00	1760.00	2060.00
FF10kTC	1585.26^{C}	154.00	1270.00	1800.00	1490.00	1730.00
FTEM	1505.00^{Bc}	155.22	1150.00	1750.00	1427.50	1612.50
FTEM10kTC	1521.88^{c}	198.77	942.00	1710.00	1490.00	1660.00

The universal type materials exhibited higher Young's modulus values than the flow type materials. A significant difference was observed between mean Young's modulus values for UTEM and Z500. Moreover, there was a statistically significant difference between Young's modulus values for FF and FTEM. A statistically significant influence of periodically alternating temperature was only observed in the case of Filtek Flow.

Tables 4 and 5 present structural parameters and goodness-of-fit to empirical data for linear regression of the properties studied as a function of filler content. Linear regressions describing the relationship between compressive strength σ_{cmax} and Young's modulus E, on the one hand, and filler content, on the other hand, are presented in Figures 4 and 5, respectively. Circles indicate the volume of compressive strength at a given filler content for the materials non-exposed to cyclically alternating temperatures, while filled circles show mean values for the same materials. The regression curve, in this case, is shown as a solid line. Triangles represent compressive strength values for specimens exposed to cyclic thermal loads, while filled triangles show mean values for the same specimens. The regression curve, in this case, is shown as a dashed line.

Table 4. *Linear regression analysis of compressive strength as a function of filler content*

	I	β_i	s_i	t_i		F_{val}	$P(F > F_{val})$	R^2
Without 10kTC	0	−233.22	76.22	−3.06	0.00318	62.03	4.028e-11	0.48
	1	8.26	1.05	7.88	4.03e-11			
With 10kTC	0	−274.98	53.87	−5.10	2.55e-06	139.20	< 2.2e-16	0.66
	1	8.75	0.74	11.80	< 2e-16			

For specimens non-exposed to the thermal fatigue test (without 10kTC), the regression model explains 48.07% of variance in compressive strength as a function of filler content (coefficient R^2). Because $P(F > F_{val}) < \alpha$ at a significance level of $\alpha = 0.05$, the null hypothesis on the lack of multiple correlation ($H_0: R = 0$, correlation coefficient insignificantly differs from zero) should be rejected in favour of the alternative hypothesis. This means that filler content affects compressive strength in a statistically significant way. For structural parameters β_0 and β_1, at a significance level of $\alpha = 0.05$, the working hypothesis should be rejected in favour of the alternative hypothesis, which means that parameters β_0 and β_1 significantly differ from zero. For the materials exposed to thermal

fatigue, a 1% increase in filler content caused an average increase in compressive strength σ_{Cmax} of 8.26 MPa. For the specimens exposed to the fatigue thermal test (with 10kTC), the model explains 65.61% of variance in compressive strength. Given that $P(F > F_{val}) < \alpha = 0.05$, it can be concluded that the correlation coefficient significantly differs from zero, and therefore filler content has a statistically significant impact on compressive strength. Also, β_0 and β_1 significantly differ from zero. For materials subjected to the thermal fatigue test, a 1% increase in filler content resulted in an average increase in compressive strength σ_{Cmax} of 8.75 MPa.

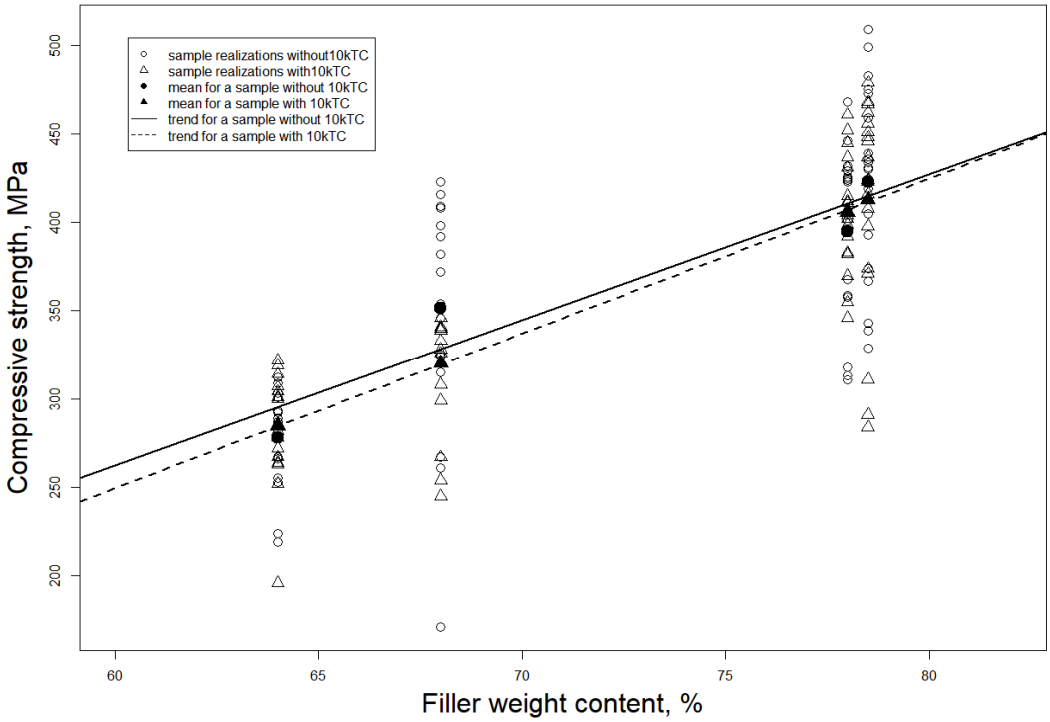

Fig. 4. Influence of filler content on the compressive strength of LC PMCCs

Table 5. *Linear regression analysis of Young's modulus as a function of filler content*

	i	β_i	S_i	t_i		F_{val}	$P(F > F_{val})$	R^2
Without 10kTC	0	−4251.21	277.44	−15.32	<2e-16	560.00	< 2.2e-16	0.89
	1	90.31	3.82	23.66	<2e-16			
With 10kTC	0	−4489.59	317.11	−14.16	< 2e-16	442.80	< 2.2e-16	0.86
	1	91.79	4.362	21.04	< 2e-16			

Linear regression analysis showed that, for the specimens non-exposed to the thermal fatigue test (without 10kTC), the model explained 89.31% of variance in Young's modulus as a function of filler content (R^2 coefficient). The null hypothesis on the lack of multiple correlation ($H_0: R = 0$, thus the correlation coefficient is insignificantly different from zero) was rejected in favour of the alternative hypothesis that filler content had a significant impact on Young's modulus. For test pieces subjected to thermal fatigue (with 10kTC), the model explained 85.85% of variance in Young's modulus as a function of filler content. Also in this case, the null hypothesis on the lack of multiple correlation was rejected in favour of the

alternative hypothesis that the impact of filler content was significant. The structural parameters of the linear models describing the relationship between Young's modulus and filler content were also significant. The experiments demonstrate that in the case of specimens non-exposed to the thermal fatigue test, a 1% increase in filler content caused an average increase in Young's modulus by 90.31 MPa. In specimens exposed to thermal fatigue, the same increase in filler content resulted in an increase in Young's modulus by 91.79 MPa, on average.

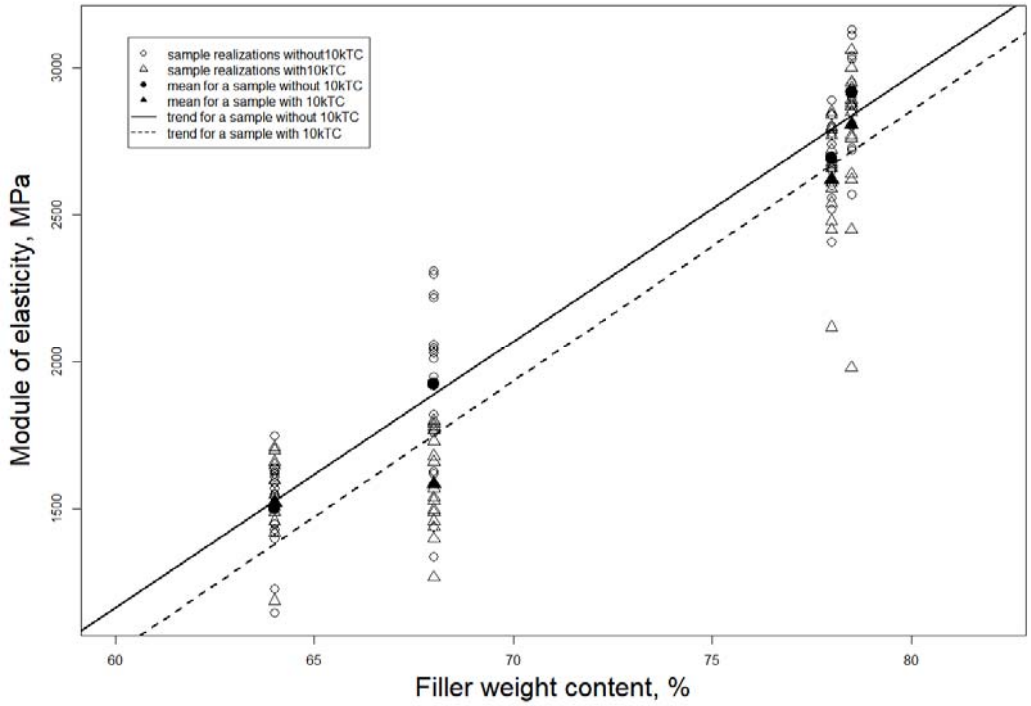

Fig. 5. Impact of filler content on Young's modulus of LC PMCCs

DISCUSSION

The results of the present study confirm the observation made by other authors that an increase in the content of filler particles in a composite leads to an increase in its compression strength [15-16]. Flow type composites contain fewer filler particles than universal type materials, and, accordingly, have a lower strength. The observed strength values for the flow type composites: flow type experimental material (FTEM) and Filtek Flow (FF), were 277.81 MPa and 351.71 MPa, respectively. To compare, the compressive strength values for the universal composites: universal type experimental material (UTEM) and Filtek Z500 (Z500), were 394.88 MPa and 423.00 MPa, respectively.

Notably, FF had a significantly higher compressive strength than the FTEM composite. This was probably related to the higher content of filler particles in the matrix of FF. The universal materials differed statistically significantly in compressive strength despite a similar filler content. This difference was related to the type and size of filler in the matrix. The influence of type of filler particles on the compressive strength of LC PMCCs has been reported by Randolph et al. [17] and Hambire and Tripathi [18], who found that zirconium oxide, and silica fillers improved the compressive strength of dental resin composites.

The impact of the size of filler particles on the compressive strength of LC PMCCs has also been demonstrated by several authors. It has been found that nano-scale filler particles

improve the mechanical properties of composites [19]. The higher compressive strength of nano-filled composites can be explained by the fact that small particles adhere more strongly to the matrix than larger ones [20]. In the present study, the universal type composites differed not only in the type of filler particles, but also in their size. The Z500 composite contained micro and nano-scale particles. UTEM, on the other hand, contained only micro-scale particles, which probably had a negative impact on its compressive strength.

The stiffness of LC PMCCs depends on the content and size of filler particles in the matrix [21]. In the present study, universal type composites had a higher Young's modulus, determined in a compression test, compared to materials with lower contents of filler particles, such as flow type composites. Additionally, statistically significant differences in Young's modulus were found between universal type composites themselves. This finding confirms the influence of particle content on the elasticity modulus. Antunes et al. [22] have shown that Young's modulus increases almost linearly with filler content. It has also been demonstrated that the increase in filler content results in an increase in stiffness, up to a certain level [23].

Operating conditions cause changes in the mechanical properties of LC PMCCs, in most cases, resulting in their gradual degradation. Exposure of a material to cyclic loads of alternating temperature can result in thermal fatigue, which, manifests, among others, as a decrease in mechanical properties. It has also been shown that good mechanical properties, obtained ad hoc, often do not correlate with the fatigue strength of materials [24].

In the present study, cyclic changes in temperature did not statistically significantly influence the compressive strength of universal type composites. In the case of flow type composites, the impact of changing temperatures was statistically significant for FF but insignificant for FTEM. Probably, FF materials underwent hydrolytic degradation as a result of water absorption [25]. Cyclic sorption and desorption of water causes strong internal mechanical stresses, initiating microcracks in the composites' structure. The effect of moisture is more destructive when internal stresses, both spontaneous and caused by factors such as thermal loads, are present in a composite with a polymer phase. Lohbauer et al. [16] have reported that hydrolytic ageing intensifies under cyclic loading. Tensile thermal stresses cause microdamage and enable penetration of water. It can be expected that water penetrating into the gap will loosen it, enlarging the defects. Formation of such cracks can be related to the exposure to temperatures above the thermal stability of the material and below the melting temperature, at which rapid stress relaxation takes place. The structure of the composite can have an impact on the development of such cracks. As it has been demonstrated by Kawakami et al. [26], propagation of cracks in composites containing pre-polymerized particles occurs at a lower range of WIN boundary values ΔK_{th} than in the case of materials containing large particles. The absorption inside LC PMCCs takes place mainly through the matrix and the so-called interphase. The absorption of liquid in this kind of materials depends on the following: type and structure of matrix, exposure time, temperature, shape of component, relative humidity, and lighting conditions. Generally, the diffusion of water particles to multicomponent materials can be described by Fick's law, which relates the degree of diffusion to time and temperature.

Water absorption depends on the content of filler particles in the matrix and their type [27]. With increasing content of filler particles, water absorption by the composite decreases; the change in the composite's properties is less important. In the case of materials with a lower filler content in the matrix, the type of filler particles has an impact on their absorption properties. The Filtek Flow composite contains porous silica particles, which increase water absorption by the material. This could have contributed to the significant decrease in the compressive strength of the composite in the accelerated aging test. A significant change in

stiffness due to cyclic thermal loading was only observed in the case of Filtek Flow. Some authors argue that water in the structure of a composite with a polymer matrix acts as a plastificator, causing stress relaxation and reduction of stiffness [28]. It is also probable that the chemical compounds contained in a water solution cause decomposition of the polymer network by degrading ester bonds [29]. It has also been proposed that moisture absorption depends on the content and type of filler particles and the type of monomers used in the production of the matrix [30]. Additionally, viscoelastic behaviour of LC PMCCs structures can also be reflected in the aging process.

CONCLUSIONS

1. The composites investigated in the present study are sensitive to cyclic thermal loading. The conditions of periodic loading of the studied materials were identical, but the observed degree of degradation was different.
2. Fatigue resistance of LC PMCCs depends on the composite's manufacturing technology, structural defects, fatigue load conditions, ageing medium, and mechanisms of destruction. It cannot be concluded that it mainly depends on the filler content.
3. Results of statistical analysis indicate that the mechanical properties of LC PMCCs deteriorate due to thermal fatigue. Strength stability is an important issue – currently not included in the standard requirements, e.g. ISO 4049 – which should be carefully considered at the stage of material design.

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