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INITIATION AND TOLERANCE OF MACRO-DAMAGE OF FIRST PLY (FBF) IN A PROCESS OF DAMAGING OF HYBRID MULTI-PLY STRUCTURES DUE TO REINFORCEMENT ARCHTECTURE

ABSTRACT

The objective of this paper was study and analysis of damaging process of multi-ply structure applied in dentistry. The aim was to analyze and experimentally evaluate tolerance of macro-damage of first ply (FPF - first ply failure) of multi-ply composite. A studied structure of composite makes a carrying structure for dental applications e.g. adhesive bridges. Influence of reinforcement structure on the residual carrying capacity of the studied multi-ply materials has been demonstrated. It has been shown that the type of fiber and fiber ribbon architecture play a major role in strength of studied reinforcements. Structures included in the study differ by the moment of macro-damage occurrence, carrying capacity and residual stiffness.

Keywords: *glass fibers, polyamide fibers, bending strength, damage tolerance, fiber reinforced dental composites*

INTRODUCTION

Composite materials due to their special properties and benefits are used in applications transferring loads in the technical systems, as well as biomechanical systems. Often inhomogeneous multiply structure materials, such as for example structural composites, including laminates are used [1]. Structural strength reflects structure resistance to damage under the influence of forces, thus strength is an ability to oppose impact of damaging forces. Knowledge on dissipative processes – main factors responsible for development of damages, enables better use of materials' strength composing structure transferring the loads [2]. Anisotropy of fibrous laminates causes the orientation of cracks and their development depends not only on the load, geometry and boundary conditions, but also on the material's morphology [3]. In order composites to maintain the highest possible damage tolerance, their damage cannot occur in a catastrophic way and should consider the largest possible volume [2] of the loaded structure.

Delamination and cracking process should take place in a progressive way. Progressive damaging process is dependant mostly on mechanical properties of structure of plies, reinforcement architecture and shape and geometry of specimens [4,5]. Process of progressive damaging is beneficial due to large amount of absorbed energy, which main mechanism is delamination [6]. In case when the active force is directed perpendicular to direction of fibers orientation, damage occurs as a result of matrix fracture, mostly in the ply, which has been stretched. Thus, it is due to the fact that in case of ceramic-polymer materials used in dentistry their tensile strength is lower than their compression strength. In the damaging process a diverse deformation of ceramic-polymer phase and reinforcement occurs, causing high local stresses at the phases' contact [7].

Complex materials, Light Cured Polymer Matrix Ceramic Composites (LC PMCCs), described in the paper, depending on the filler structure, can be classified according to [8] as particle, grain, powder composites. The dispersed phase of composites are particles with similar three spatial dimensions. Due to shape, such fillers are sometimes called as 3D [8]. PMCCs are characterized by multiphase structure, commonly consist of two basic phases: polymer (organic) constituting approx. 20-40% of material's volume [8], ceramic (non-organic) constituting approx. 60% of material's volume [8] (up to 70-80 wt%), and photoinitiators. The organic phase is usually based on the light cured methacrylate resins. Among them the following can be listed: bisphenol A glycidyl methacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA), and polycarbonate dimethacrylate (PCDMA) [9]. Strength characteristics and elasticity depend on reinforcing mechanisms of LC PMCCs. To these materials the elastic-plastic model is not applicable. The composites are characterized by low capacity to irreversible deformations, they are brittle [10,11,12]. Additionally, it was stated that the composites are not resistant to the fast growth of cracks [13]. They are characterized by limited resistance to dynamic loads, in particularly impact loads. On the other hand, it is required that LC PMCCs, in operating conditions, cycling loads, were characterized by durability and stability [14]. Therefore, to increase the applicability, reinforcements in form of long fibers were introduced.

In structural composites studied in this paper, the most common are reinforcements in a form of fibers ribbons connected without weave and in a form of fabric of different weave [7]. The fiberglass in a form of ribbons, with specific weave, which are used in dental treatments have good dielectric properties and very good wettability, enabling strong connection of reinforcement with polymer phase.

A strength of single fiber depends on its diameter, its increase results in increase of defects occurrence probability [15]. Disadvantageous property of fiberglass is high sensitivity to water impact, requiring covering with protective plies. Polyaramide fibers of aromatic polyamide are characterized by low density, good mechanical properties and high corrosion and chemicals resistance. A disadvantage of this material is low bending strength [16]. They have a high specific modulus, much higher from the specific modulus of fiberglass [17,18].

Application of reinforcement in a form of polyaramide fibers ribbons in order to reinforce complex materials LC PMCCs is impeded, usually a modification of surface with hydrophobic hydrophilic structures coupling agent is applied (e.g. silanization). Additionally a process of silanization is more impeded due to lack of free OH groups on the surface of polyaramide fibers to react with silan [19].

In case of multi-ply structure of laminate 'strength' term is not used. Such structure is a complex structure consisting of single plies, and the same as in case of each engineering structure only carrying capacity can be used. The same approach seems appropriate for medical structures [20]. Term 'strength' is reasonable in case of single-ply composite, which is a composite of a structure similar to laminate [21]. In the studies the assumption has been made that crack tangential to cross-section of one ceramic-polymer ply (non-destructive damage related to loss of strength by first ply) does not cause a loss of carrying capacity by the structure and enables further transfer of biomechanical loads, thus use of residual carrying capacity in operation. This assumption can base on the conclusion from [22] among the others. Usually, as a limit of carrying capacity is considered a point where damage occurs due to significant delamination or breakage of reinforcing fibers (critical failure - destructive damage).

The objective of this paper was to study and analyze damaging process of multiply structures applied in dentistry.

EXPERIMENTAL

The objects of the studies were composite structures applied in biomechanical systems, specifically in dentistry systems as a replacement for biomechanical structures of natural tissues. The aim of using dentistry bridges is reconstitution of physiological functions. Composite structure which is the subject of the analysis is a carrying support for permanent composite applications e.g. crowns, bridges (fig. 1), refills, temporary and permanent splinting of twisted teeth. Stresses generated in a composite carrying structure of composite foundation such as among the others of bridge, are caused by transfer of occlusive forces.

In order to evaluate the influence of material and architecture of reinforcement on the damage, modeling conditions (simplifying) were assumed with regards to real bridge structures or splinting of dental arch. For studies rectangular specimens reinforced with long fibers with different architecture of weave were made as per presented scheme in fig. 2.

To make composite structures ceramic-polymer composites were used, fiber glass and polyaramide fibers. In the studies commercial used materials were applied. It was assumed that study of commercial material will be more useful. Characteristics of composite plies are shown in table 1. The specimens were treated with halogen lamp (HAL) and diode lamp (LED) with source diameter of 10 mm. Specimens were cured at three points, starting from the specimen’s centre. Exposition time of each exposed zone was 40 s.

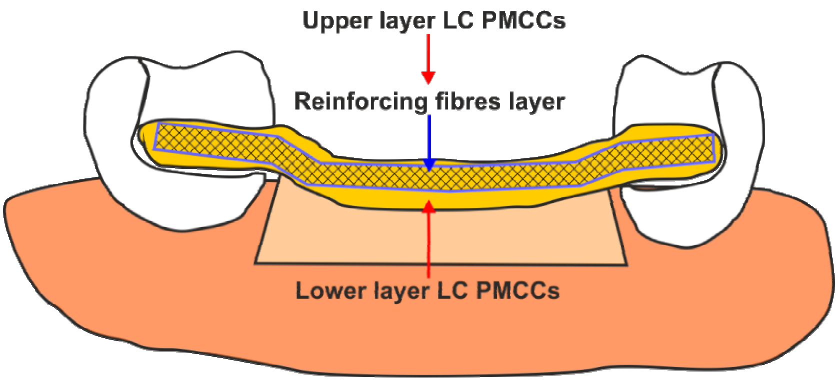


Fig. 1. Scheme of carrying foundation of adhesive bridge (own work according to LFS Arkona)

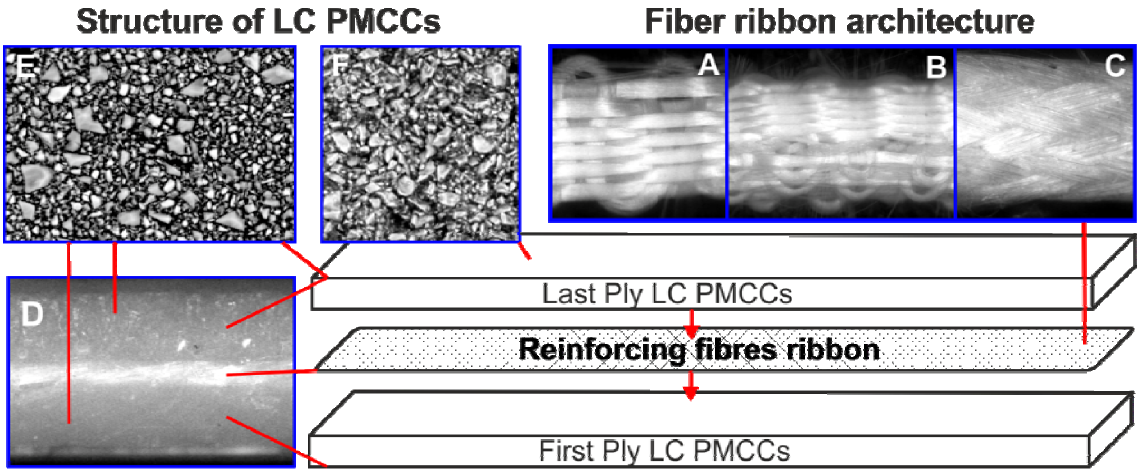


Fig. 2. Arrangement of plies in specimens used for studies and architecture of reinforcement weave: a) single ply fibreglass weave, b) three ply fibreglass weave, c) "braid" weave of polyaramid fibres, d) SEM image of plies arrangement in composite, e) SEM image of microhybrid composite structure, f) SEM image of hybrid composite structure

Table. 1. *Specification of materials used in the study*

Type of Ply Structure				
Light Cured Polymer Matrix - Ceramic Composite (LC PMCC)	Hybrid Polymer Matrix - Ceramic Composite (HC)		Micro Hybrid Polymer Matrix - Ceramic Composite (mHC)	
	Type of filler (79% weight)	Inorganic minerals fillers with average particle size of 0,6 μm	Type of filler (78% weight)	Baria-alumina-silica glass, igneous silica, titanium dioxide
	Manufacturer	Kerr	Manufacturer	experimental
	Fiber ribbon structure			
Fiberglass	Fiberglass with single ply weave (1LR_GF) Fig. 2a		Fiberglass with triple ply weave (3LR_GF) Fig. 2b	
	Manufacturer	Polydentia	Manufacturer	Polydentia
Poly-aramide fibers	Poly-aramide fibers of braid weave type (BR_P-AR) Fig. 2c			
	Manufacturer	experimental		

Study method

Studies of reinforced light cured polymer matrix ceramic composites (LC PMCCs) were conducted using test and specimen parameters with external dimensions complying with technical standard ISO 4049 [23]. The study based on strength test at conditions of three-point bending was applied, which has a model nature and does not fully reflect load conditions at clinical conditions [24]. However, it is a codified method and study according the standards enables comparison. Specimens used in the study were made in a form of prismatic cuboid beams with dimensions of *b = 2 mm, d = 2 mm, c = 25 mm* (fig. 3). Strength studies were applied on Zwick/Roell Z100, traverse speed was *0,5 mm/min*, support span *L = 20 mm*. Radius of supports and loading pin was *1 mm* (fig. 3). In the study a load cell Xforce (Zwick/Roell) with nominal range of *500 N* was used.

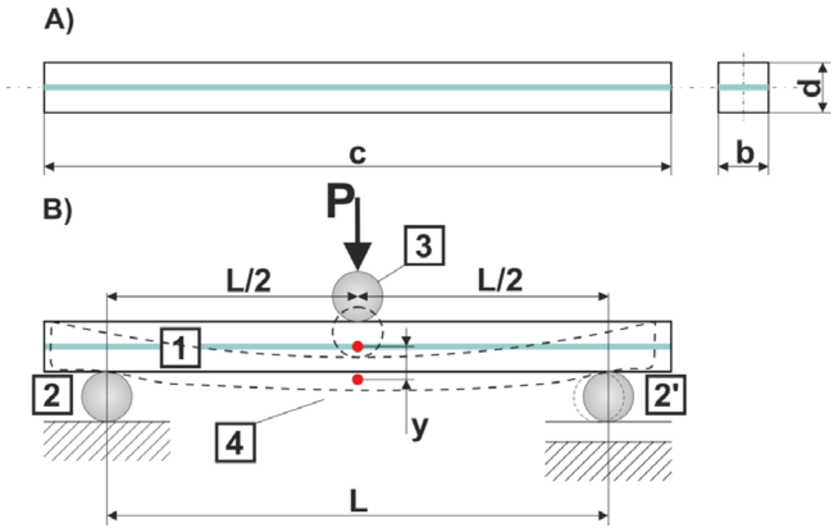


Fig. 3. Dimensions of the study specimen (A and setup for strength studies at three point bending (B): 1-specimen, 2- support, 2'-movable support, 3 – loading pin

Description of damage propagation after macro-damage of first ply was based on damage process according to generalization by Camanho and Davila [25]. At figure 4 a model shape of bending characteristics which served as basis for analysis is shown. Figure shows specific points of damage process of first ply LC PMCCs (1 - 1'), takeover of the load by the other plies (2) and point of carrying capacity loss as a result of catastrophic damage (critical damage) (2').

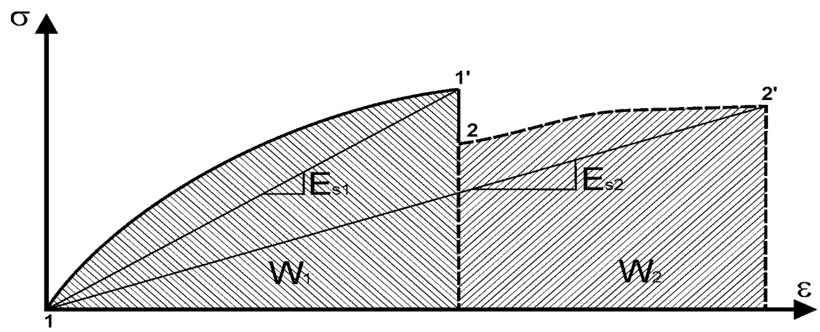


Fig. 4. Stress characteristic as a function of three-ply structure deflection (PMCCs – fibreglass/polyaramid - PMCCs) showing progressive damaging process (description of the marking in the text)

Test characteristics were used to determine characteristic parameters for carrying capacity and damaging. Bending damaging stresses of first ply LC PMCCs were estimated based on the following formula:

$$\sigma_1' = \frac{3PL}{2bd^2} \text{ [MPa]} \quad (1)$$

where:
P - active load during the test [N]
L – supports span [mm]
b - specimen width [mm]
d - specimen thickness [mm]

Whereas secant elasticity modulus was estimated based on the following formula (fig. 4):

$$E_S = \frac{\Delta\sigma}{\Delta\varepsilon} \tag{2}$$

where:
$\Delta\sigma$ - stress growth [MPa]
$\Delta\varepsilon$ - corresponding $\Delta\sigma$ deflection growth [%]

Surface area under the curve was calculated σ-ε, obtaining the measure of damage energy. In energetic approach, work of external forces causing deflection equals internal energy of the material's deflection. It was assumed that the damage of first ply is related to its cross-cracking and linked to step growth of deflection of the bended beam. Work of macro-damage of first ply W_1, as an energetic measure, gives a basis for evaluation of total load size and damaging deformation of first ply. W_1 work was determined based on the below equation:

$$W_1 = \int_0^{y_1} F\,dy_1 \tag{3}$$

where:
F - load at test of specimen [N],
y_1 - beam deflection at the moment of macro-damage of first ply (First Ply Failure) [mm].

As it has been already mentioned as a result of macro-damage of first ply a partial loss of carrying capacity occurs. Propagating damage has a progressive character. Residual carrying capacity is maintained, which can be expressed in energetic terms. The work force on the deflection is a result of bending of other undamaged plies. Calculations were conducted for the part of curve σ-ε from the moment of takeover of load by undamaged plies, thus from point 2 to 2' in figure 4.

$$W_2 = \int_{y_2}^{y2'} F\,dy_2 \tag{4}$$

where:
F – load during the test [N], y_2 – beam deflection at the moment of load transfer by the other plies [mm], $y_{2'}$ – beam deflection at the catastrophic damage [mm].

Mechanical strength of first ply LC PMCC specifies material behaviour under influence of quasistatic loads. One of the major factors influencing strength of LC PMCCs is size and distribution of random manufacturing deviations [26], it is worth mentioning that dental applications are manufactured manually. Due to statistical scattering the results were subjected to Weibull's analysis. The aim of analysis was to determine the equivalent statistic strength (σ_e) of first ply, equal to scale coefficient from Weibull's distribution. Scale parameter understood as specific strength value corresponds to *63,2%* cases of damage of first ply. Additionally Weibull's modulus (*m*) was determined - shape distribution parameter. Weibull's modulus can be considered as a uniqueness strength parameter (dispersion) of first ply. Procedure of Weibull's analysis was described in work of [27].

For clinical success of reconstruction a stiffness of carrying structure of bridge especially splinting of dental arch is essential. There are no unequivocal recommendations describing parametrically stiffness of such structures. In the literature there is only information about recommended minimum value of elasticity modulus bending of dental composites, which should be not less than 10 GPa [28]. Degradation of stiffness can be caused by not fulfilling biomechanical and physiological functions. It can be assumed partial deterioration of stiffness as a result of first ply damaged most strenuous ply LC PMCCs, can be not crucial and will not have clinical importance. Therefore, one of the objectives of the presented studies was to determine a degree of tolerance of damage of first ply due to residual stiffness until the moment of damage. In work of [29] it was proposed that in case of acting of bending loads to specify damage by changing stiffness, which depends mostly on elasticity modulus. Hwang [30] suggested the following damage function formula:

$$D = 1 - \frac{E}{E^*} \tag{5}$$

where:
E – current value of tangential modulus, E^* - initial value of tangential elasticity modulus.

Based on analysis presented in works of [31,32] a modified form of damage function was adopted, based on elastic modulus, primary E_{s1} and secondary E_{s2}:

$$D = 1 - \frac{E_{S2}}{E_{S1}} \tag{6}$$

Modulus was used to determine damage functions values of the additional specimens. Following descriptive statistics of D parameter were calculated.

A total loss of carrying capacity was related to complete destruction (catastrophic). For the need of analyses of phenomena, in the presented study, a compilation of total work curves to damage W, which corresponds to sum of W_1 and W_2 works is shown. W work was presented as a function of angle of beams deflection. Similar approach is presented by [29], which also relate to specimens deflection.

RESULTS

Results of studies of stress work on deflection to macro-damage of first ply (FPF) (W_1) and work to damage (W_2) are presented on box-plots shown in figure 5. In figure 6 a box-plot of damage parameter with regards to stiffness loss due to damage of first ply (FBF) is presented.

Figure 7 shows results of Weibull's analysis of damaging load of first ply (FPF). Approximation of probability of damage based on non-parametric equation (7) is given in figure 7. A linear approximation of results of studies enabled determination of regress equations.

$$P_f = \frac{i - 0.3}{n + 0.4} \tag{7}$$

where:
i - value specifying location of strength value in a ranked set,
N - number of specimens in set.

Ranking of measurements results was based on setting strength results from the lowest to the highest and assigning numerical values from the real numbers set from *1* to *n*, thus *i=1,2,3,....., n*.

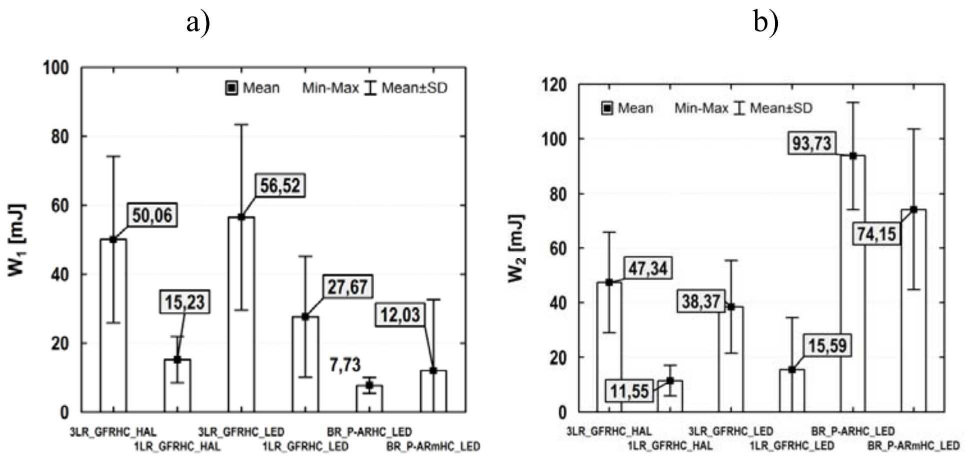


Fig. 5. Box-plot of work of macro-damage of first ply (W_1) and work to damage (W_2)

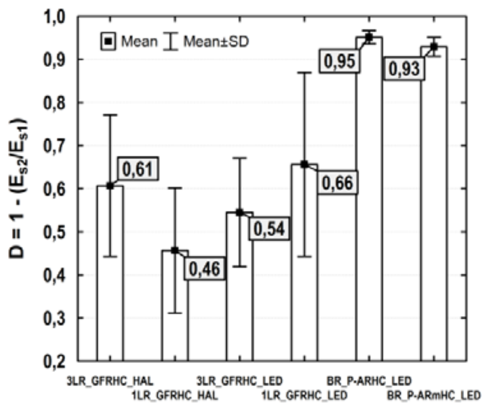
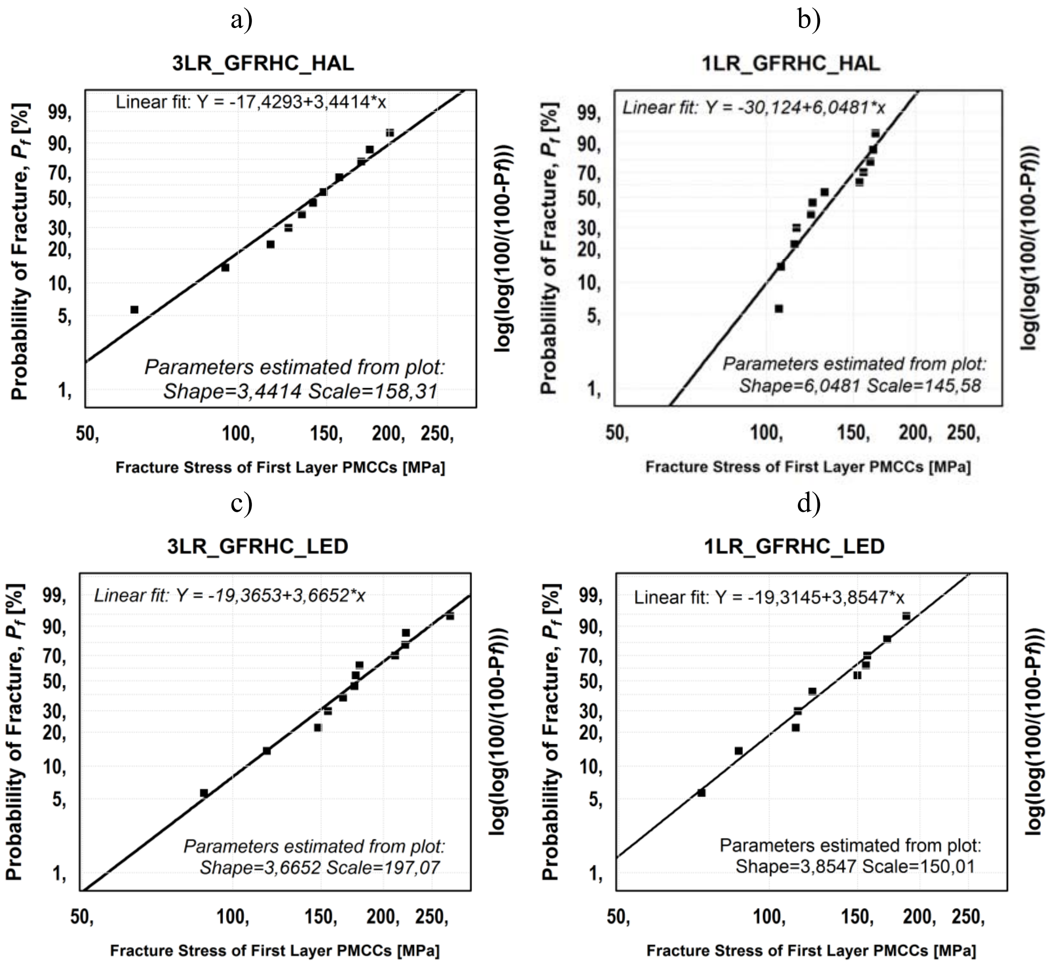


Fig. 6. Degradation degree of stiffness due to macro-damage of first ply (FPF)

DISCUSSION

Reinforcement with fibers can ensure residual carrying capacity and stability at clinical conditions in real dental applications. Cooperation between reinforcing fibers and PMCCs, thanks to which it is possible to transfer loads and deformations by the composite, is a basic property of analyzed composites. Very important due to durability of dental applications is nature of this cooperation after macro-damage occurrence of firs ply (FPF). In [22] it was shown that dental bridge after occurrence of macro-damage can survive for some period of time under the influence of decreased load level. Damage of the ply can occur as a result of damage of supporting ceramic-polymer ply, caused by acting of shear stresses in ply plane or forces perpendicular to direction of fibers arrangement. As it has been shown strength and stiffness characteristics, show dependency from reinforcement mechanisms, which was also stressed in works of [31]. PMCCs do not apply to ideally elastic-plastic body mode. They are characterized by low ability to irreversible deformations. Additionally use of fillers do not limit fragility of dental composites, they are much more fragile than metals [32,33,34]. It can reflect relatively low load value needed to initiate damage of first ply. Additionally, it has been found that these composites have low resistance to rapid growth of cracks [35], causing almost immediate cross-over cracking of first ply (macro-damage FPF) after reaching critical stress. Initial defects can also have significance, occurring in a process of ply structure creation by the dentist. In work of [36] the initial non-critical technological defects of structure are given, occurring in fiber reinforced composites: insufficient or uneven resin curing, incomplete wetting of the fibers through resins - Poor adhesion of resin fibers, air bubbles, voids - fibrous area not filled by polymer phase - ceramics, delamination - flat discontinuities between plies; loose fiber ends; damaged fibers; -matrix cracks, and other resulting from their own observations: shrinkage resulting in high self-stress in the ceramic polymer phase, gradient of ceramic-polymer ply properties caused by the polymerization method.



e) f)

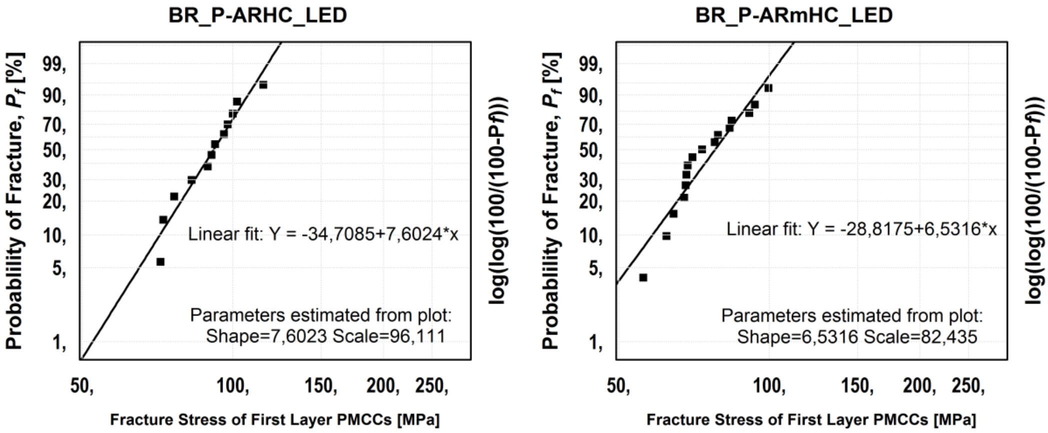


Fig. 7. Weibull's grids describing stresses damaging first ply FPF

Initial structure defects can have impact on energy level needed to initiate damaging process and tolerance of evolving macro-damage up to damage. Damage can also occur in intermediate ply between fiber and matrix, so called damage at phase interface [28] (fig. 8a). Damage in this area can be initiated before breakage of first ply and even as a result of polymerization shrinkage. After damage of first ply PMCCs, delamination process dominates (fig. 8B), which as shown in the presented studies is dependent on fibers architecture.

a) b)

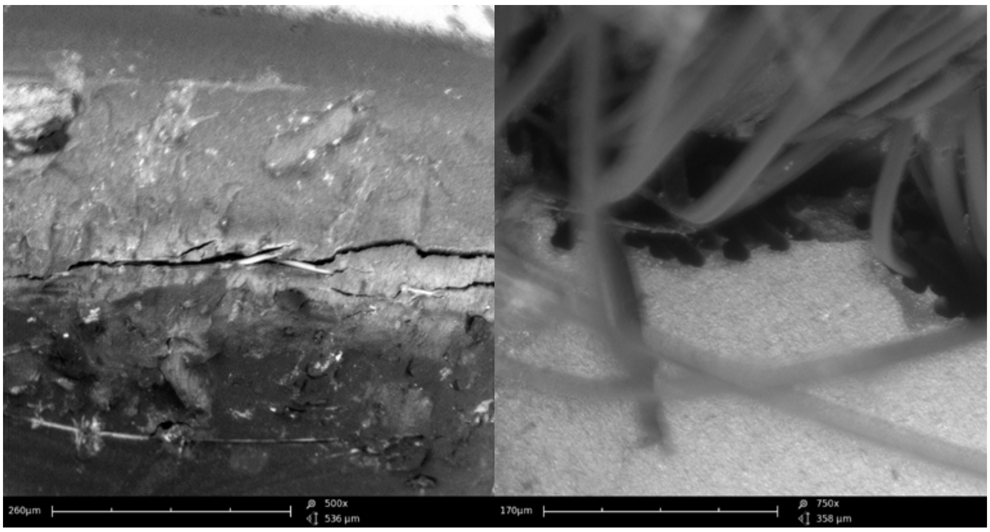


Fig. 8. Damage at phase interface between ceramic-polymer and reinforcement (a), delamination of fibres in a damage process (b)

It appears that delamination of LC PMCCs plies from reinforcement depends on two factors, adhesion force and possibility of penetration of non-cured composite between the fibers, into the reinforcement structure. Tight weave of bride type (fig. 9A) does not favor penetration and limits binding of plies, mostly to adhesive forces (fig. 8b). It is also worth mentioning that in the presented bending studies, the ration between specimens height to width *L/d* was similar to *10*. Therefore in the presented test the impact of inter-ply shear was not significant [37].

Adhesive forces of fiber ribbon, soaked technologically with resins which due to ability to infiltrate improves binding of the structures, are large and favor maintaining of residual carrying capacity. In case of one-ply reinforcement of fiberglass with loose woven weave (fig. 9C), PMCC penetrated deeper into the reinforcement (fig. 10a). At this point it can be referred to results of force work at deflection W_1 and W_2. In case of reinforced structure with braid weave, W_1 and W_2 works differ significantly. W_2 (fig. 5b) work is couple of times higher than W_1 work needed for initiation of macro-damages (FPF) and their value depend mostly from significant delamination resistance (fig. 11a). Other dependency was observed in case of one-ply reinforcement of fiberglass (1LR_GFRHC), in this structure delamination after macro-damage progressed with lower resistance (fig. 11b).

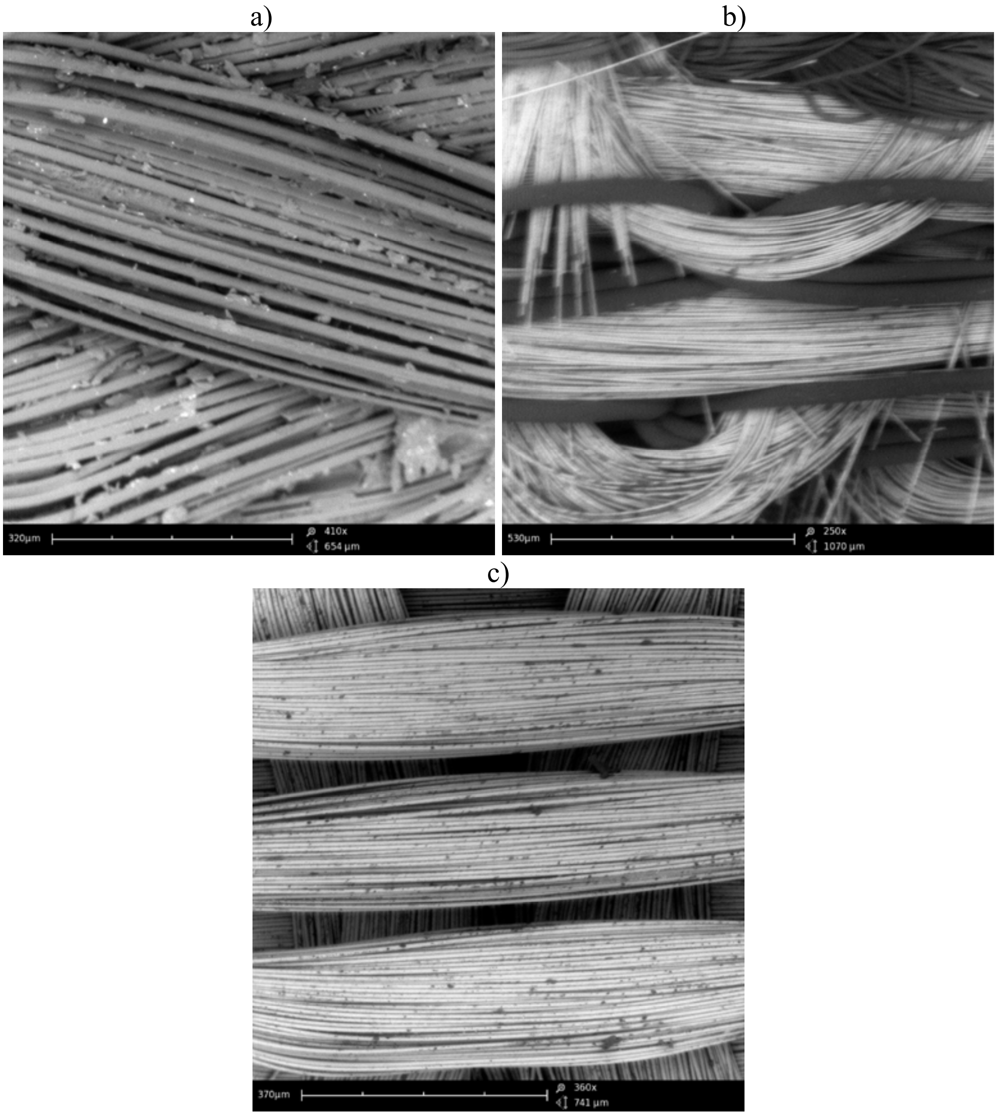


Fig. 9. Polyaramid fibres with bride weave after loosening from PMCC (a) ply, three-ply weave of fibreglass (b), single-ply weave of fibreglass (c)

Equivalent static strength σ_e of first ply of structures BR_P-ARHC_LED and BR_P-ARmHC_LED was 96,11 and 82,43 MPa, respectively (fig. 7). Obtained values are significantly lower from the obtained reinforced structures with fiberglass. Values of W_1 work are also the lowest for reinforced structures with braid weave fibers. Lower strength of first ply PMCC can be a result of local stress pile up reflecting maladjustment of reinforcement rigidity and PMCC (fig. 10b). Significant is also a possibility of mutual movement of plies with different formality under the load, braid tight weave (fig. 9A) limited such possibility. Additionally high stiffness and good adhesion of this reinforcement caused concentration of compressive stresses in upper ply PMCC and next its breakage (fig. 11), which consequently was a reason for loss of carrying capacity and damage of the structure. From the information presented in the work of [38], follows that in order to use strength of fiber ribbon, ceramic - polymer ply must show good deformability. In case of reinforcement with braid architecture, possibility of deflection of PMCC ply was difficult due to incompatibility of mechanical properties with reinforcement properties. It seems that loose three-ply weave of fiberglass (fig. 2b and fig. 9b) facilitated the process, which reflected highest σ_e and W_1 values.

a) b)

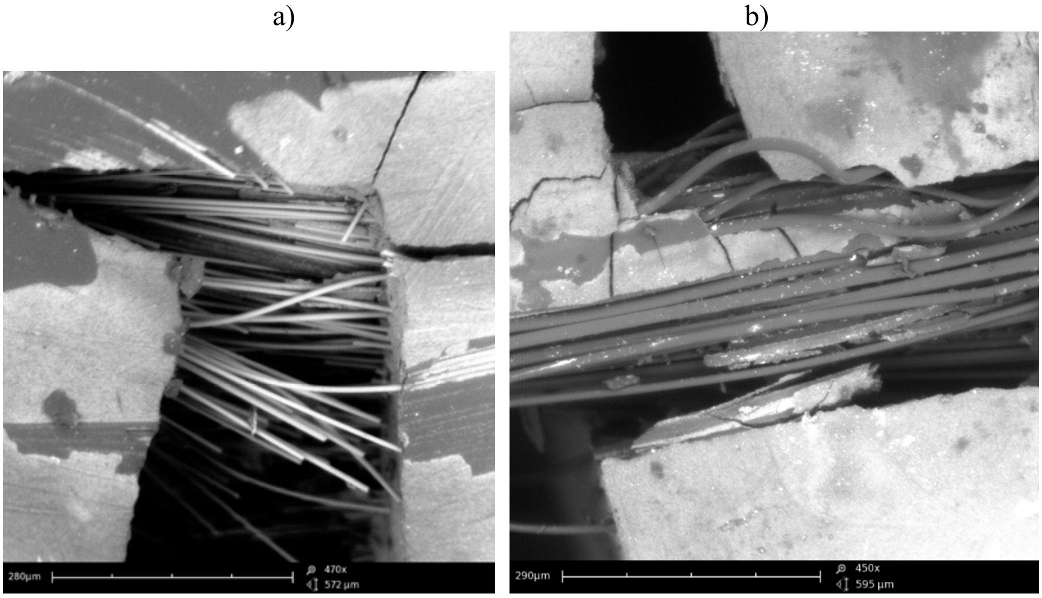


Fig. 10. Structures breakthrough 1LR_GFRHC (a) and BR_P-ARHC (b)

Structures BR_P-ARHC_LED and BR_P-ARmHC_LED, were characterized by initial stiffness similar to other studied plied structures. Mean elasticity modulus E_{s1} of all studied specimens was 7,27 GPa, whereas BR_P-ARHC_LED and BR_P-ARmHC_LED, 6,26 GPa and 8,81 GPa, respectively. Mean residual modulus E_{s2} of all studied specimens was 2,22 GPa, whereas BR_P-ARHC_LED and BR_P-ARmHC_LED, 0,31 GPa and 0,62 GPa, respectively. Thus, degradation of stiffness shown as damage parameter D (fig. 6) of structures reinforced with polyaramide fibers with braid weave was the highest. Reinforced structure with single ply weave of fiberglass, cured with HAL lamp was characterized by the lowest degradation of stiffness at level of $D = 0,46$ and at the same time not higher than initial stiffness.

a) b)

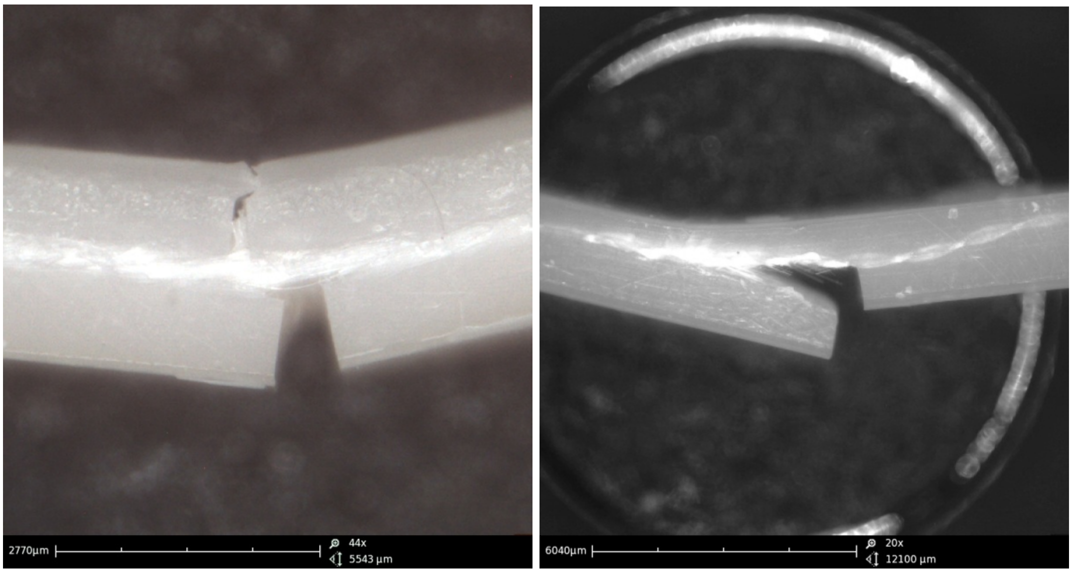


Fig. 11. Specimens scraps a) BR_P-ARmHC, b) 1LR_GFRHC

CONCLUSIONS

The following conclusion has been formulated based on the conducted studies:

1. Reinforcement fibers architecture has a crucial role in strength of dental applications.
2. Damaging process of studied multiply structures is gradual and progressive, it propagates from micro-damages through transverse cracks - tangential to the cross-section in PMCC ply, delamination up to destruction, sometimes catastrophic damage appearing as complete loss of carrying capacity caused by fibers breakage (in the presented studies only in case of fiberglass).
3. Despite similar values of total stress work on the deflection to damage, structures included in the study differ by the moment of macro-damage occurrence, carrying capacity and residual stiffness.

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