VYSOKÉ UČENÍ TECHNICKÉ V BRNĚ
BRNO UNIVERSITY OF TECHNOLOGY

FAKULTA CHEMICKÁ
ÚSTAV CHEMIE MATERIÁLŮ
FACULTY OF CHEMISTRY
INSTITUTE OF MATERIALS SCIENCE

POKROČILÉ VRSTEVNATÉ KOMPOZITY PRO APLIKACE VE STOMATOLOGII

ADVANCED LAYERED COMPOSITES FOR DENTAL APPLICATIONS

AUTOREFERÁT DIZERTAČNÍ PRÁCE

AUTOR PRÁCE Ing. Zbyněk Šedivý
AUTHOR

VEDOUCÍ PRÁCE prof. RNDr. Josef Jančář, CSc.
SUPERVISOR

BRNO 2013

KLÍČOVÁ SLOVA

vrstevnaté kompozity, mechanická odezva, homogenizace, částicové kompozity, vláknové kompozity, minimálně invazivní stomatologie, test na tříbodový ohyb

KEYWORDS

layered composites, mechanical response, homogenisation, particle filled composites, fiber reinforced composites, minimally invasive dentistry, three-point bending test

Table of Contents

1 INTRODUCTION

The presented work deals with layered composites used in dentistry. This type of composites provides new possibilities for clinical dentistry. It combines usually one or more layers of particle reinforced/filled composites with one or more layers of fiber/fabric reinforced composites. This concept offers exceptional esthetics important in dentistry along with high strength and stiffness and consequently it provides dentists with a clinically reliable, versatile and cost effective alternative to traditional dental metal alloys and ceramics.

The major topics of presented work are:

- to review the basic material and clinical aspects of layered composite,
- to review strategies for multi - scale modelling of layered composite,
- to investigate effects of microscale and mesoscale structural features of model, layered composite superstructures on stiffness and stress distribution.

2 EXPERIMENTAL

2.1 MATERIALS

There were following materials selected for the preparation of the model test specimens:

(This is not full description, commercial names are used, however, detailed composition must be briefly described –type of resin, filler loading, manufacturer´s declared properties)

- BOSTON CB, manufacturer Arkona - Laboratorium Farmakologii Stomatologicznej, Lublin, Poland - crown and bridge composites (high-viscous light curing particle filled composited with silica particles and methacrylate based resin),
- BOSTON FLOW, manufacturer Arkona - Laboratorium Farmakologii Stomatologicznej,, Lublin, Poland - flowable composites (low-viscous light curing particle filled composited with silica particles and methacrylate based resin),
- light curing methacrylate based resin (50% BIS-GMA, 50% TEGMA),
- DENTAPREG PFU, manufacturer ADM a.s. Brno, Czech Republic, unidirectional prepregs (S and E glass fibers, methacrylate based resin),
- DENTAPREG SFM, manufacturer ADM a.s. Brno, Czech Republic, multidirectional prepregs (E glass fibers, methacrylate based resin).

2.2 METHODS

2.2.1 Structural Analysis

In order to characterize materials used in mechanical analysis several techniques of structural analysis were employed:

2.2.1.1 Thermogravimetric analysis - TGA

TGA was carried out in order to determine fibre / particle volume fraction in respective composites.

2.2.1.2 Differential scanning calorimetry - DSC

DSC was carried out in order to determine degree of conversion (DC) in cured matrices of particle filled composites as well as unidirectional prepreg, which were used in the layout of layered composites examined in mechanical tests.

2.2.1.3 Scanning electron microscopy - SEM

SEM observations were carried out in order to provide morphological analysis of microstructural features, which are of relevance to interpretation of micromechanical processes which influence macromechanical behavior of the specimens.

2.2.1.4 High speed camera monitoring

To record the visual effects elucidating mechanisms causing fracture under three-point static bending, high speed digital camera monitoring was used.

2.2.2 Mechanical Analysis

2.2.2.1 Dynamical mechanical thermal analysis - DMTA

DMTA was carried out in order to evaluate visco-elasticity of respective materials by determining storage modulus, loss modulus, and loss tangent.

2.2.2.2 Static three point bending test

Static three point bending tests was conducted in order to obtain comparative data of flexural properties of layered composites. The geometry of the test was relevant for clinical applications (width of the specimen) and ensured that shear during bending could be omitted (aspect ratio of the specimen equal 30).

The measurements were performed on specimen of beams of 20 mm (width) x 20 mm (depth) x 70 mm (length) where span of the support was 60 mm.
The test arrangement is illustrated schematically in Fig.1.

2.2.2.3 Finite element analysis

Finite element analysis was used as a complementary tool in this work. We have used the calculation protocol proposed by Jan Pěnčík [68] for ANSYS software package for fiber reinforced structures. The chosen model includes influence of large displacements, and rotation of the finite elements.

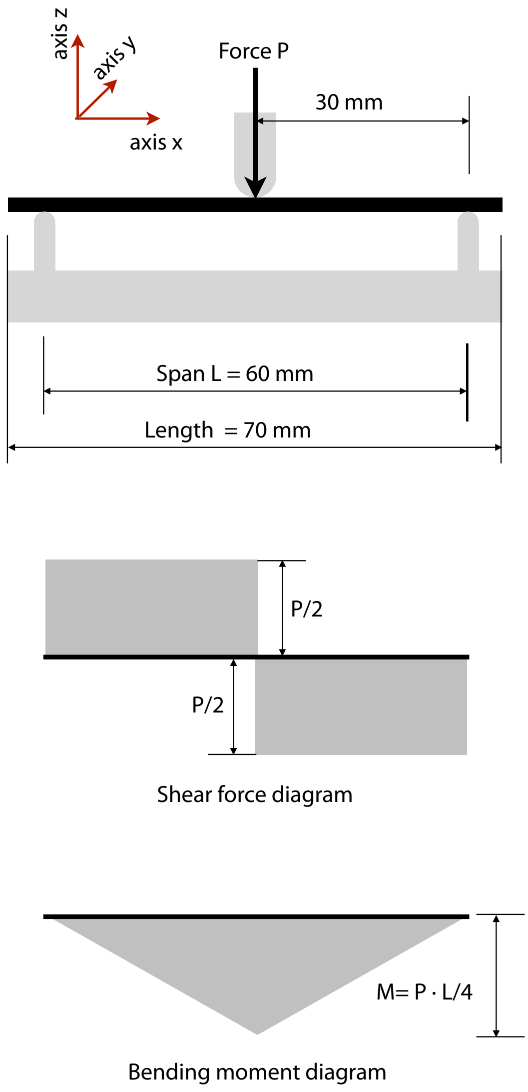

***FIGURE 1**: Three point flexure test together with shear force and bending moment diagrams*

2.2.3 Specimen schedule

Several types of beam layout were prepared according to the schedule in the TABLE 1:

***TABLE 1**: Specimen schedule*

Type of the specimen	Particulate composite (PFC)	Fiber reinforced composite (FRC)	Interlayer
Components (mono-layered beams)			
F	Low viscous PFC (FLOW)	None	None
C	High viscous PFC (CB)	None	None
P	None	Unidirectional strip PFU	None
S	None	Multidirectional strip SFM	None
Combinations (bi-layered beams)			
F1P	Low viscous PFC (FLOW)	Unidirectional strip PFU x 1	Direct contact
C1P	High viscous PFC (CB)	Unidirectional strip PFU x 1	Direct contact
C1PI	High viscous PFC (CB)	Unidirectional strip PFU x 1	BisGMA-TEGMA
C2P	High viscous PFC (CB)	Unidirectional strip PFU x 2	Direct contact
C3P	High viscous PFC (CB)	Unidirectional strip PFU x 3	Direct contact
C4P	High viscous PFC (CB)	Unidirectional strip PFU x 4	Direct contact
C1S	High viscous PFC (CB)	Multidirectional strip SFM x 1	Direct contact
C2S	High viscous PFC (CB)	Multidirectional strip SFM x 2	Direct contact

3 RESULTS AND DISCUSSION

3.1 STRUCTURAL ANALYSIS OF LAYERED COMPOSITES AND THEIR COMPONENTS

3.1.1 Thermogravimetric analysis

The results obtained from the TGA measurements are in good agreement with the manufacturer's data – see Figure 2.

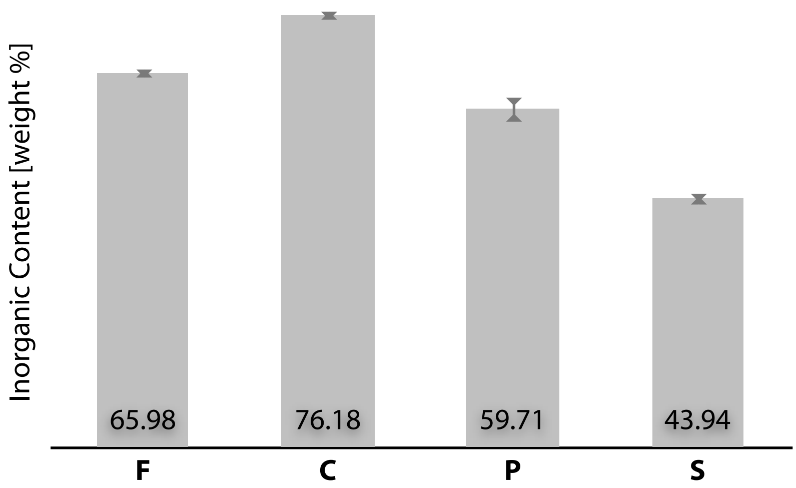

FIGURE 2*: Contents of inorganic material in components of layered composites*

3.1.2 Differential scanning calorimetry

Output data testified that there were no transitions present during irradiation. Based on this fact it can be considered that all specimens were fully cured during preparation – see Fig.3.

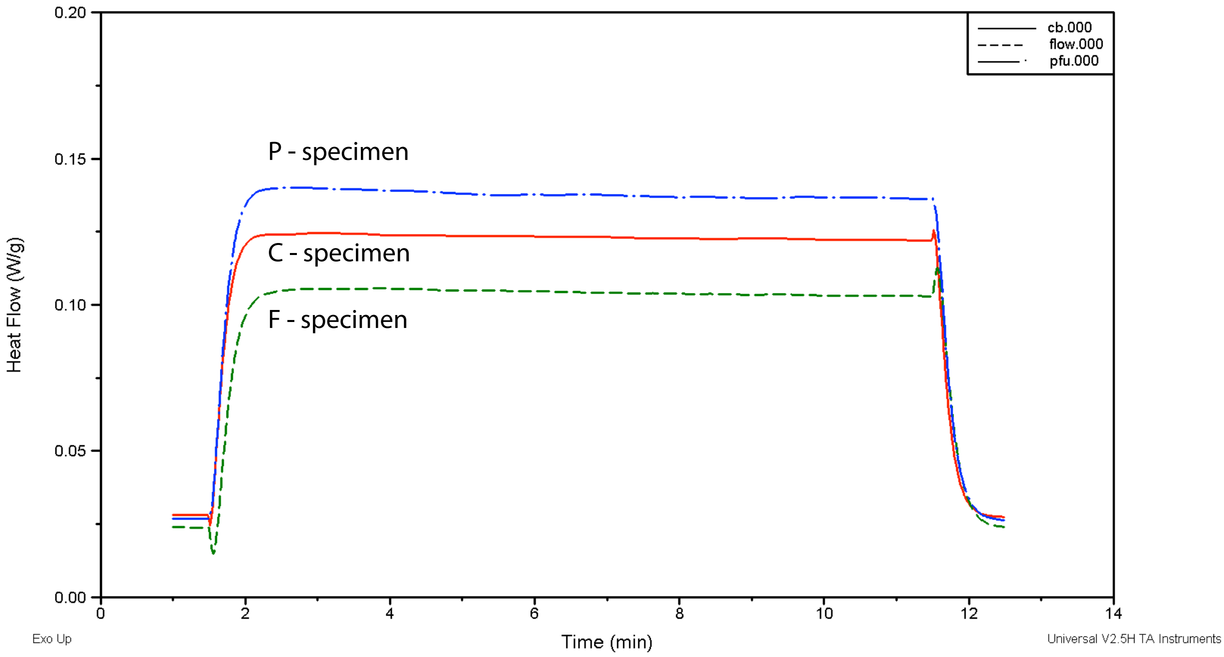

FIG. 3*: Heat flow of cured components of layered composites*

3.1.3 Scanning electron microscopy

Scanning electron microscopy was used in order to analyse visual features of deformed specimens after being subject to the static three-point bending test. Following types of beams specimens were analysed:

- F – specimen
- C – specimen
- P – specimen
- S – specimen
- C1P – specimen
- C1S - specimen

Significant visual features are shown in the following series of SEM pictures in different magnification.

Beams of particulate composite of type F and C failed during static three-point test in the brittle fracture manner (see Figure 4). The fracture surfaces were smooth and in a larger magnification silica filler particles embedded in the resin became visible. There are no major differences between the micro-structure of F and C beams as their components are very similar. On the C specimen fracture surface defects of internal pores are visible. Their diameters vary around 10 μm and they are typical for nanofill and microhybrid composites. These flaws are originated during the manufacturing process [53].

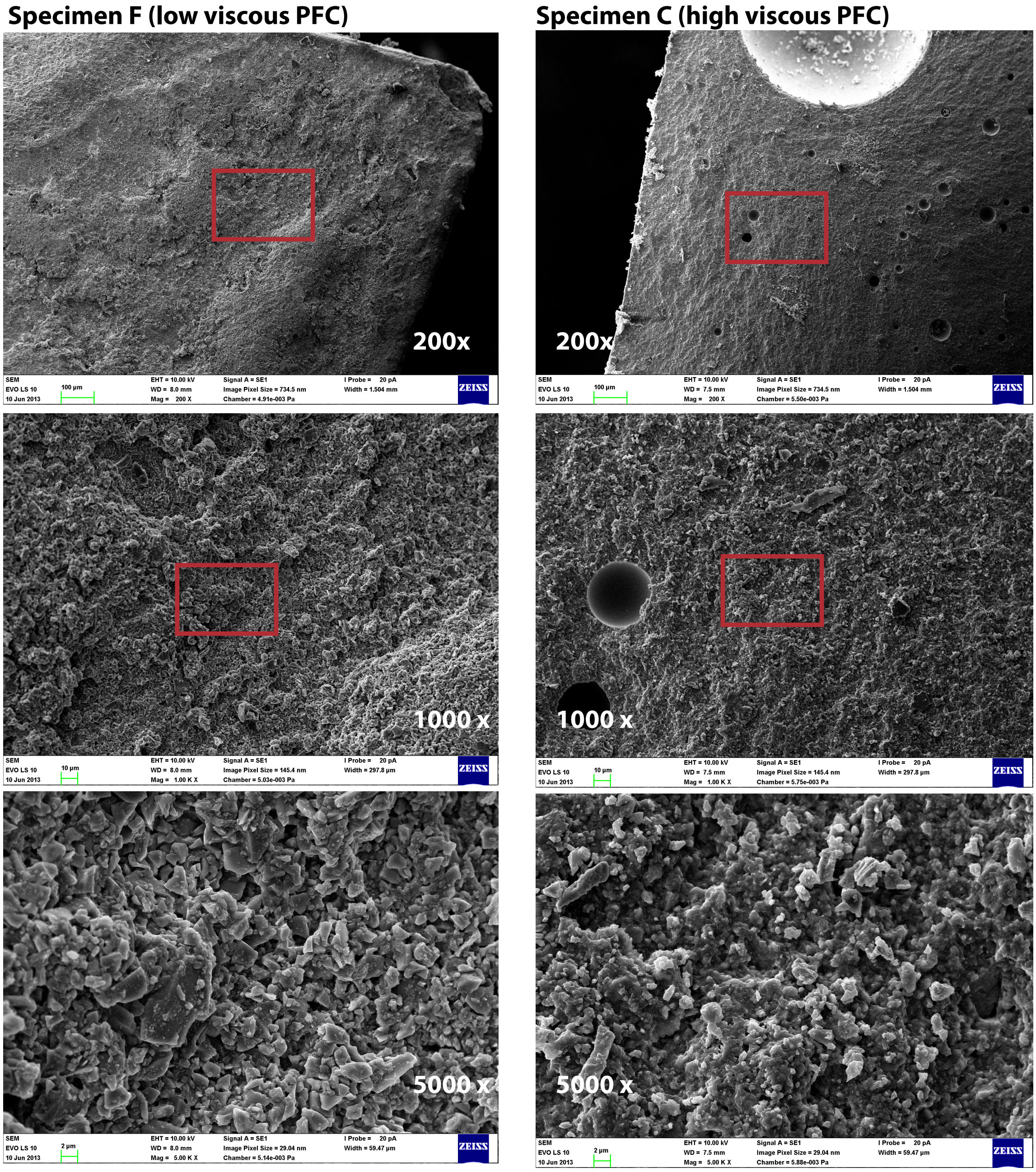

FIGURE 4*: SEM of fractured particulate filled composites F and C*

P composite displays non-uniform dispersion distribution of fibres across the cross section of a single pre-impregnated strip (see Figure 5). Consequently resin rich domains in the PFU strip specimen are founded. When deformed in three-point bending, the failure of the specimen is caused by several types of events:

- The fibers break due to growing tension in a range where they are not surrounded by the matrix. In the range where they are embedded in the matrix, the matrix bridges the fractured part and transmits the load to other part of the fibre or to adjacent fibres. This event reduces tension strength [67].

- The fibers loss their stability (microbuckling) due to growing compression in a range where they are not surrounded by the matrix. In the range where they are embedded in the matrix, the matrix bridges the buckled part and transmits the load to other part of the fibre or to adjacent fibres. This event reduces strength in compression [65].
- The matrix cracking due to raising tensile stresses. This event reduces stiffness of the beam. It also increases areas where fibers are not embedded in the matrix.
- Fibre de-bonding (pull-out) occurs when bonding strength is exceeded due to local stress concentration [60]. The consequence is reduced stiffness of the beam.

All types of events develop gradually and in parallel in the areas of local micro-structural geometry discontinuities resulting in local stress concentration. As these micro-events propagate more individual fibres or bunches of fibres detach from the resin matrix the loss of stiffness becomes evident and finaly the beam losses its strength due to the buckling of fibres in the compressed part of the cross-section of the beam. Growth of the stress in the part of the cross-section which is still able to carry the load leads to the progress in detachment of the fibres and the matrix and finally the beam fails due to the disintegration of its micro-structure.

When considering failure of S beam (see Figure 5) it is necessary to take in account the structure of the lamina. Uncured multi-directional lamina has well defined outer geometry due to mechanical bond of braided yarns. This structural feature leads to different behavior of the multi-directional lamina within the layred composite compared to uni-directional lamina. Outer geometry of uncured uni-directional lamina changes when the strip is built in a layered composite. Individual fibers disperse across boundaries of the layers and make the transition between layers gradual.

Gradual transition sustains larger shear load between layers compared to a steep transition. In steep transition between layers smaller area than in gradual transition resists the load. It is in accordance with experimental results published in [54].

The failure of the S beam is controlled by delamination of individual laminae due to the inter-layer shear stress which exceeds the adhesion between the layers.

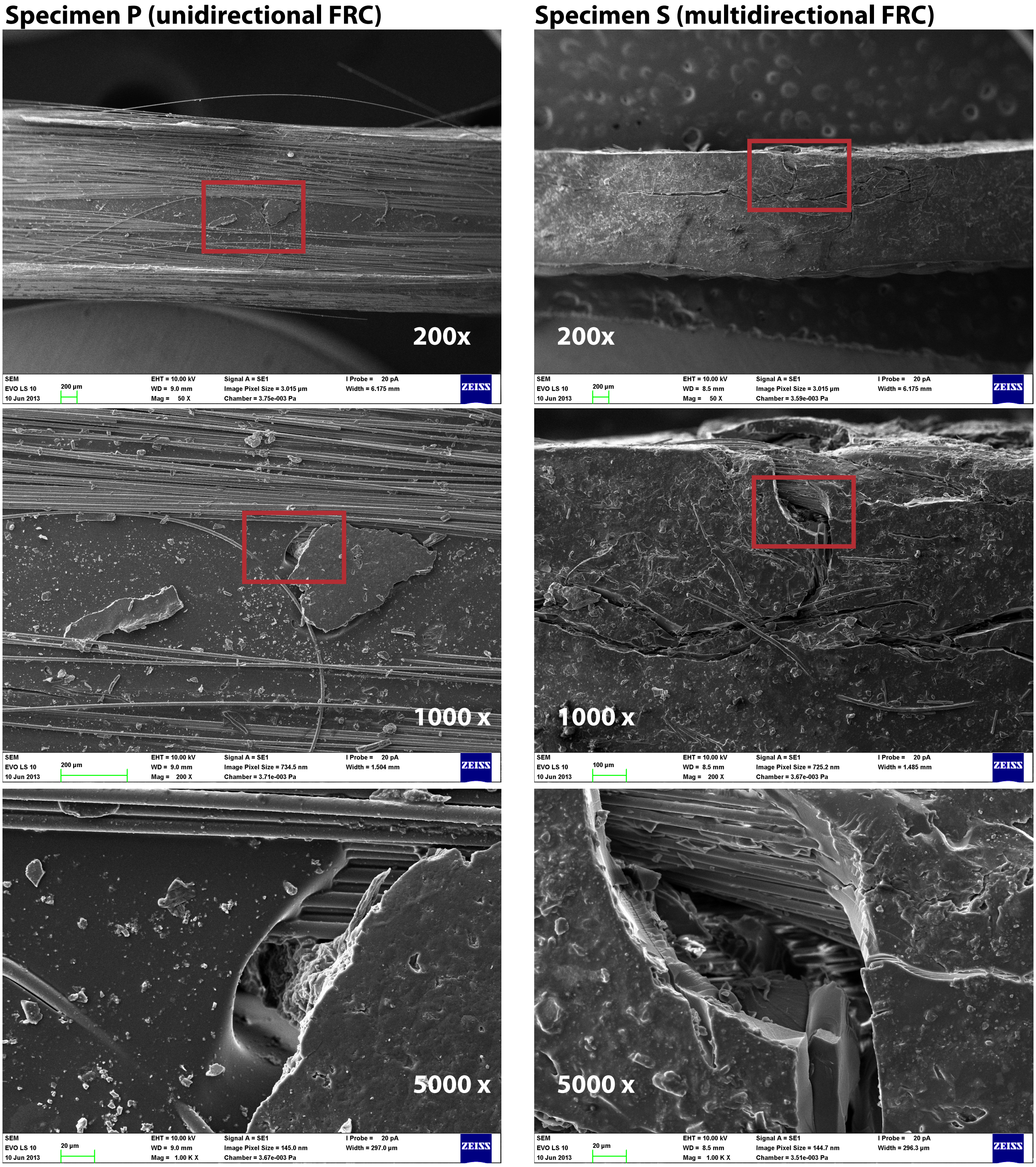

FIGURE 5: *SEM of fractured fibre reinforced composites P and S*

Layered beam C2P based on the combination of two uni-directional FRC layers (P) and high viscous particulate filled composite (C) exhibits fracture surfaces in the PFC layer (see Figure 6). Fibres in the lower layer preclude cracks propagation from the PFC layer. The normal stress parallel to longitudinal axis of the beam caused by bending in FRC layer leads gradually to debonding of individual fibres followed by tensile rupture.

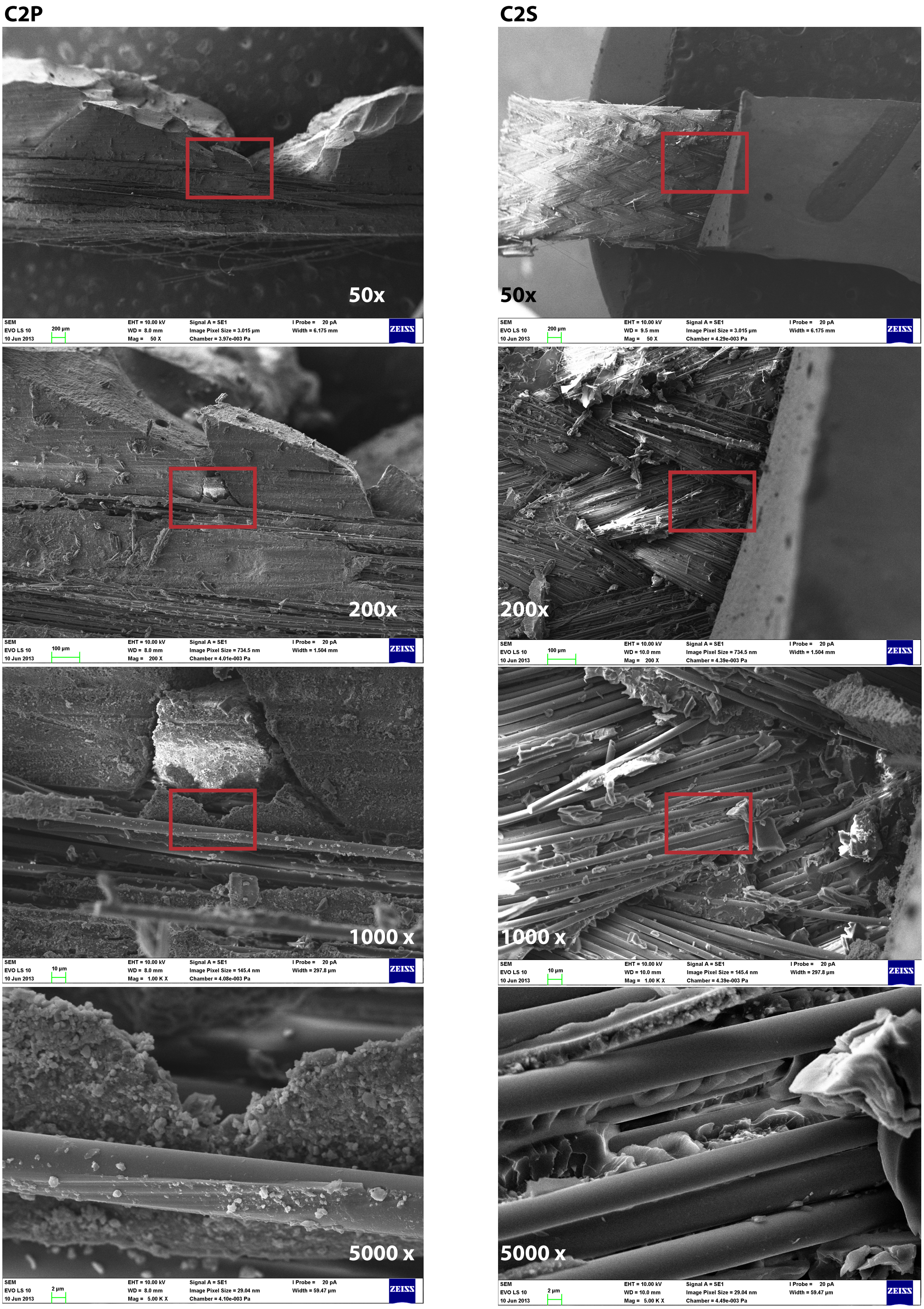

***FIGURE 6**: SEM of fractured fibre reinforced composites P and S*

It is apparent that the microstructure of all types of specimens exhibits large number of different inhomogeneities such as voids, fibers misalignment, imprefections in fibre-matrix interface caused by fabrication process. These microstructural features causes an appreciable scatter in parameters of mechanical

Layered beam C2S combining layers made of two multidirectional FRC strips (S) and high viscous particulate filled composite (C) displays failure brittle fracture of PFC followed by de-bonding of the FRC layer adjacent to CB layer and finally delamination of individual laminae in the FRC layer.

Output of finite elements analysis depicts the distribution of normal stress in the stage of initiation of FRC and PFC layers delamination – see Figure 7.

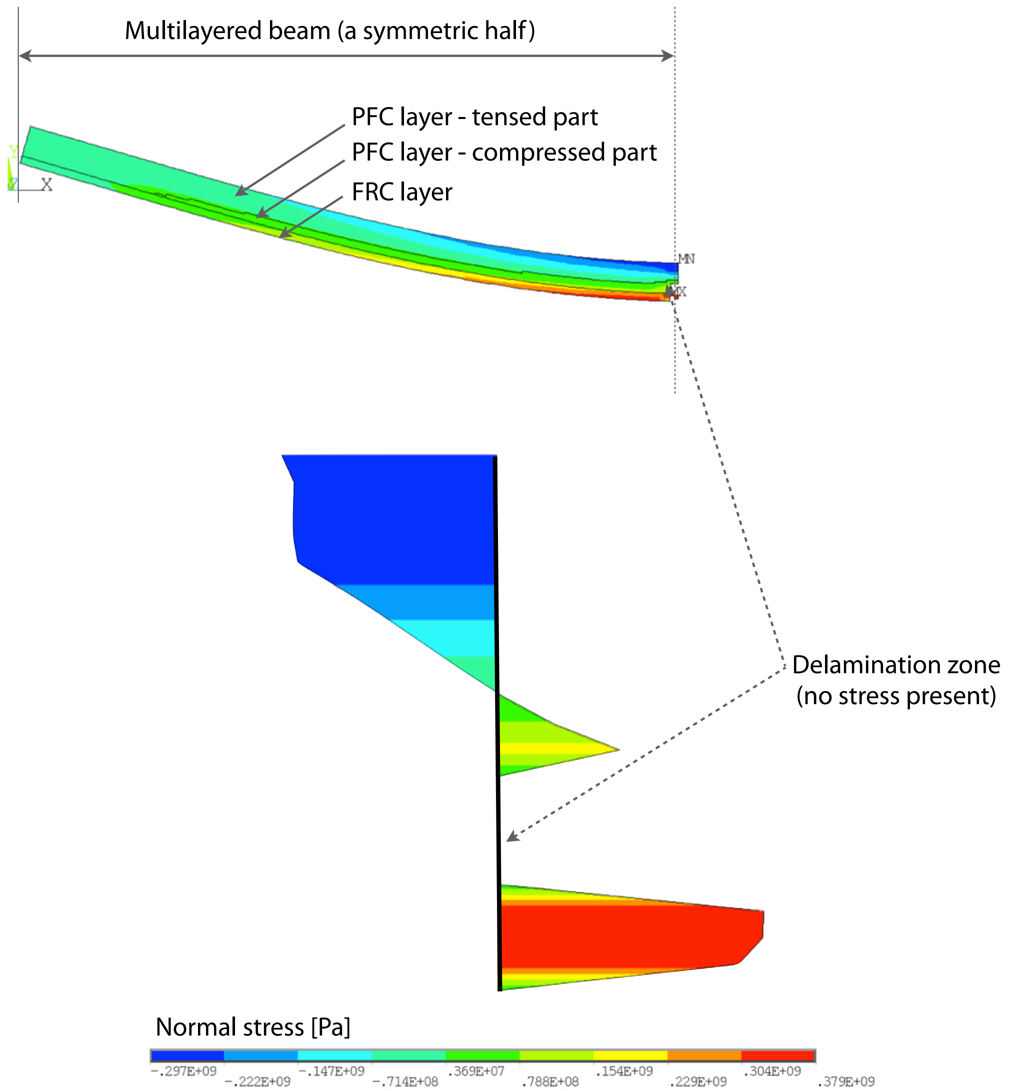

FIGURE 7: FEM analysis of normal stress distribution during initiation of delamination of the layers in bi-material beam

3.1.4 High speed camera monitoring

High speed camera recorded static three-point test of selected type of specimen in order to visualize deformation events in the specimen and relate them to the characteristic points of load - deflection curve.

Figure 8 shows different modi of the failure of the specimen P (a) and the specimen C4P (b).

P beam failed due to separation fibres from the matrix in the compressed part of the cross section followed by buckling.

C4P beam failed due to separation fibres from the matrix in the tensed part of the cross section followed by gradually rupturing the individual fibres in tension.

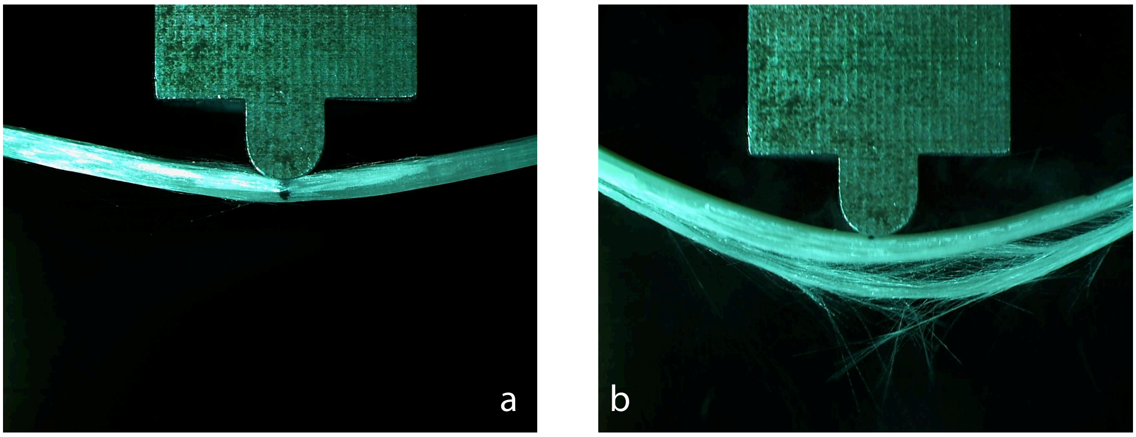

***FIGURE 8**: Modi of failure of the specimens P (a) and C4P (b*

3.2 MECHANICAL ANALYSIS

3.2.1 DMTA tests

All types of beams investigated exhibited relatively large ability to store energy from the deformation (quotient of E_{ST} > 99.5% of E_{DMTA}) i.e. prevailing mode of the response was elasticity - see Fig.9.

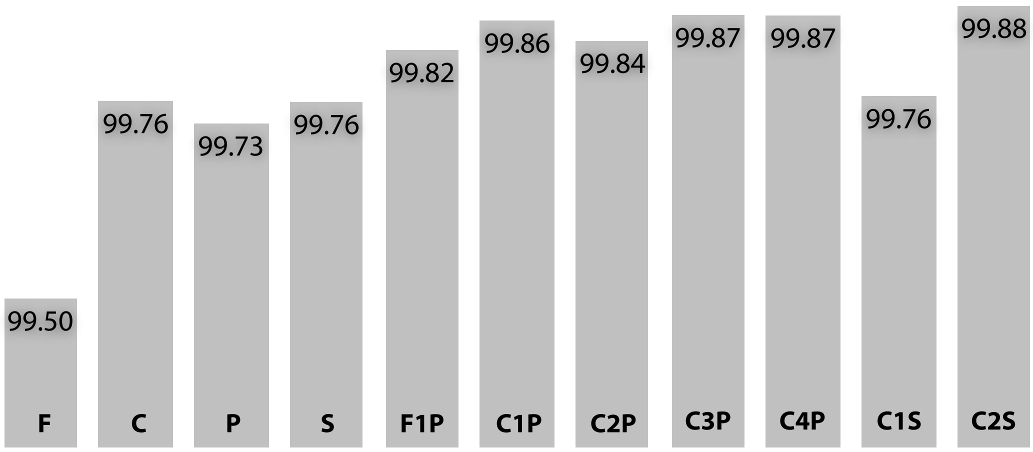

***FIGURE 9**: Quotient of E_{ST} [% of E_{DMTA}]*

3.2.2 Patterns of mechanical response on static three-point bending

Several characteristic values were derived from the data obtained during tests such as stiffness, modulus of elasticity and characteristic stresses in the cross-section of the specimen.

Two forms of load-deflection diagrams were observed:

- 1 - Smooth curve of the relation demonstrates continuous deformation without sharp changes within internal structure of the specimen. A single peak of the load – deflection curve is followed by a catastrophic reduction in load (stress) in one or more steps. The respective SEM images indicates the failure mode of this type of specimen.
- 2 - The complex curve with multiple local extremes (peaks) demonstrate multi-steps changes within internal structure. Multiple peaks are indicative of subcritical cracking [57] and they are accompanied by stress redistribution which leads to the next peak on the curve. Outputs of finite element analysis of the specimen F1P illustrates how the normal stress is redistributed during deformation when subcritical cracks propagate – see Figure 10 a,b.

Only a part of the range of data was included in the stiffness analysis in specimens with complex load-deflection curve. This range begins at the start of the curve and ends when the first peak occurs. When deriving characteristic (maximum) stresses the range of the curve where maximum load occurs is taken into consideration.

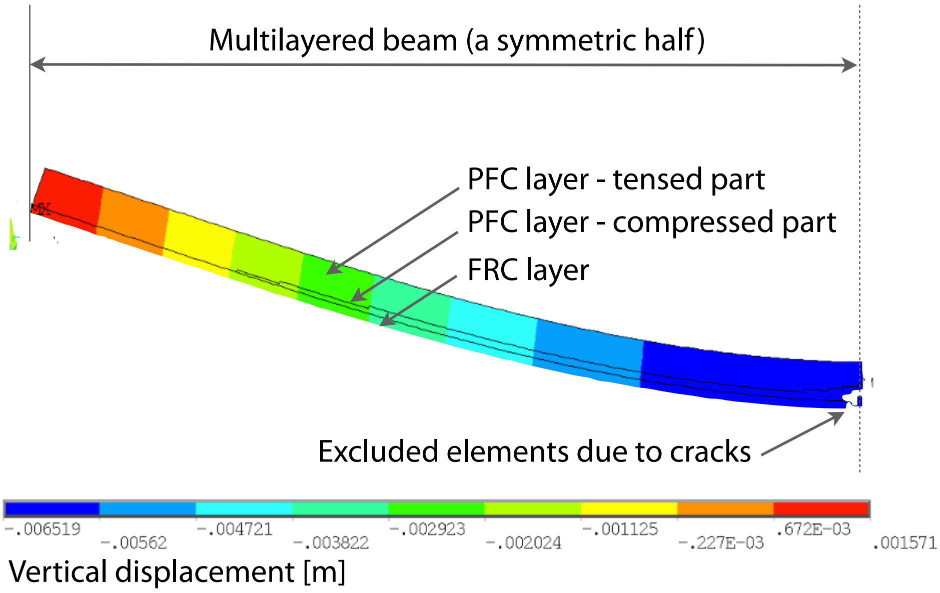

FIGURE 10a*: FEA of vertical displacements in specimen F1P during subcritical cracks propagation*

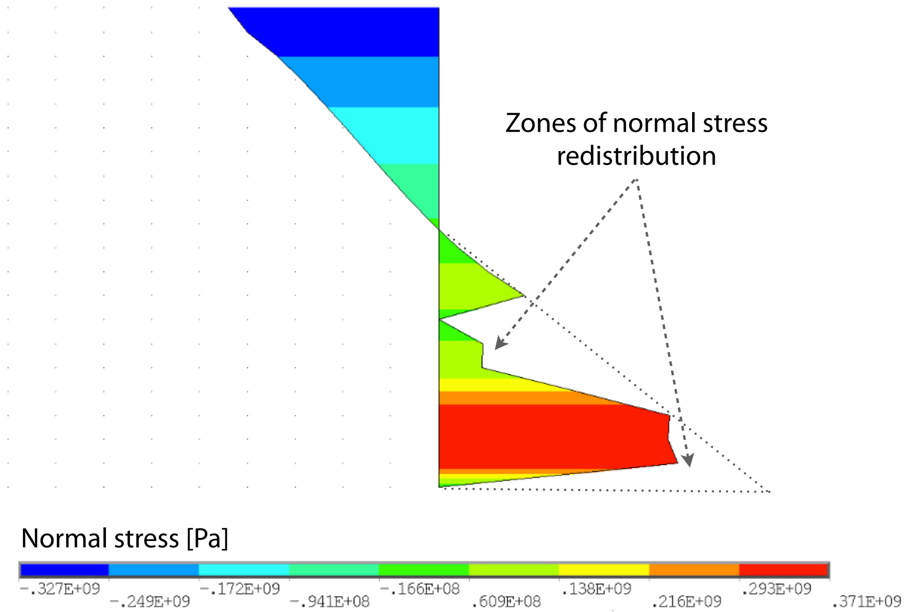

FIGURE 10b*: FEA of normal stress distribution in specimen F1P during subcritical cracks propagation*

3.2.3 Stiffness in the initial range of deformation

3.2.3.1. Influence of filler content of PFC on stiffness

When considering filler content of particulate filled composite in a layered beam it influences the resistance of the beam against deformation. Layered beam of the same sequence of layers but with higher filler content of PFC layer displays higher stiffness compared with that with lower filler content of PFC – see Figure 11.

A filler content influences the stiffness in accordance with the principle of equivalent homogeneity. Both theories (effective medium theory and equivalent inclusion theory) lead to the same qualitative output: the higher content of the component with the higher value of certain parameter brings the higher effective value of the respective parameter of the final composite. Silica filler has higher modulus of elasticity than the resin and in accordance with the principle of equivalent homogeneity the higher content of silica filler brings higher stiffness to the final composite. This feature of the micorstructure PFC layer is demonstrated at macroscale in the higher stiffness of the layered composite.

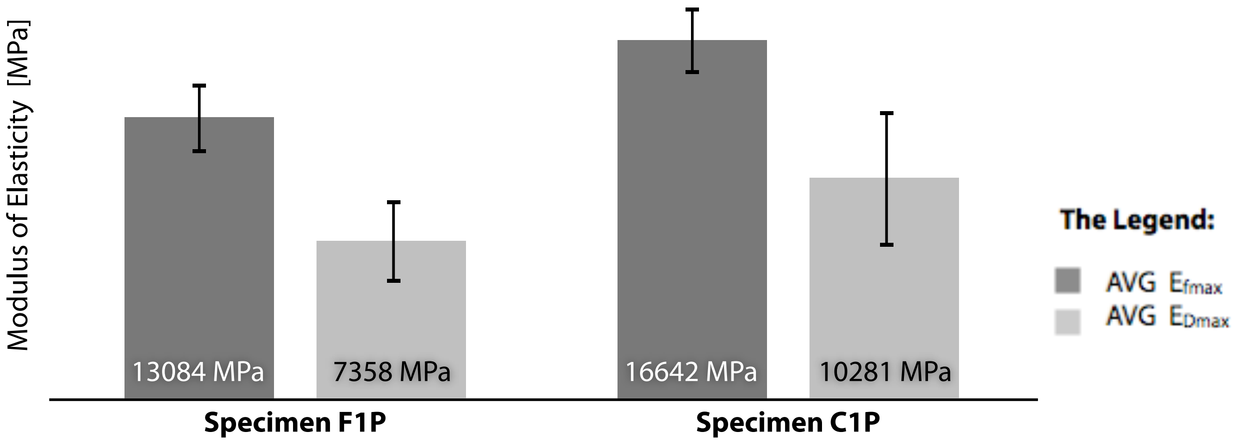

FIGURE 11*: Influence of filler content of PFC on stiffness*

3.2.3.2. Influence of the reinforcement on stiffness

In a layered composite FRC, a layer can be seen as a reinforcing part of the superstructure. The microscale elastic properties of FRC lamina are well mathematically described and bending tests provided relevant data which can be considered as effective properties of FRC as well.

Due to the homogenization of FRC layer it is possible to treat FRC as a homogenous layer with effective elastic properties given obtained by calculations or by experiment. We use experimental data obtained from three – point tests in this work. In this respect the measure of the reinforcement is the volume content of FRC layer in the specimen measured by the portion of FRC layer on the cross section area of the beam. We call this parameter in further text as a rate of the reinforcement and it is related to specific type of FRC .

As seen on Figure 12 this parameter – the rate of the reinforcement - strongly influences the stiffness of the layered beam. Higher portion of FRC layer on the cross section area (rate of the reinforcement) brings higher stiffness of the beam.

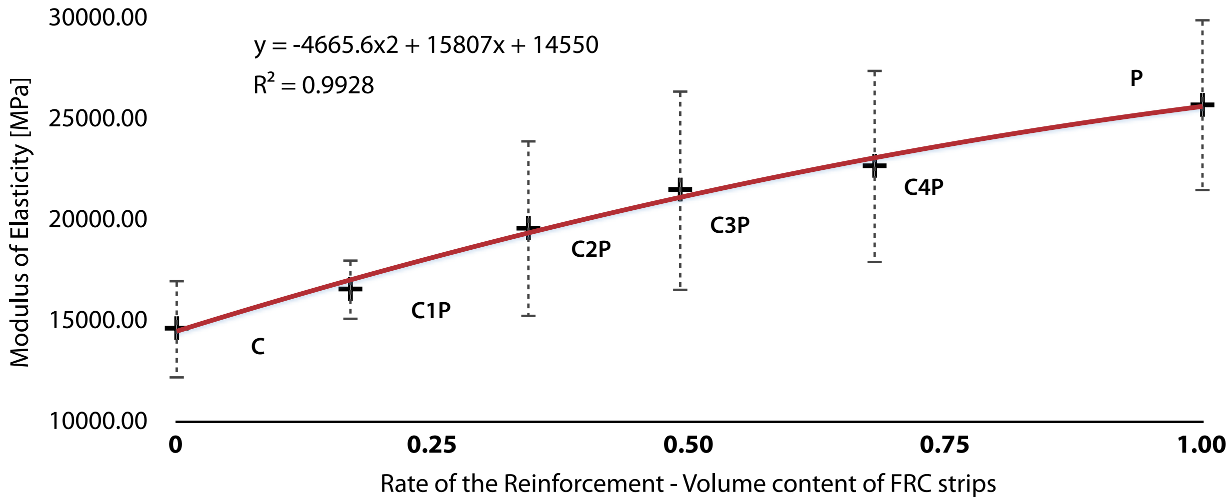

***FIGURE 12**: Influence of the rate of the reinforcement on stiffness*

3.2.3.3. Influence of the interlayer on stiffness

There was no impact of different interlayer designs (resin interlayer between FRC and PFC layers or no interlayer) in our test observed in our test – see Figure 13. It indicates that adhesion between layers does not necessarily influence the resistence of the beam against deformation which is in good agreement with the fact that the resin used for interlayer has lower modulus of elasticity than other constituents (2408 MPa ± 176).

There was neither negative influence observed because the thickness of the interlayer was ~ 0.01 mm (based on SEM image analysis) and the volume content of the resin in the beam could be neglected.

However, this result should be considered in the context of a fiber architecture of FRC. When the longitudinal shear strength between the layers controls the overall mode of failure than the presence of interlayer may positively contribute to the stiffness of the beam.

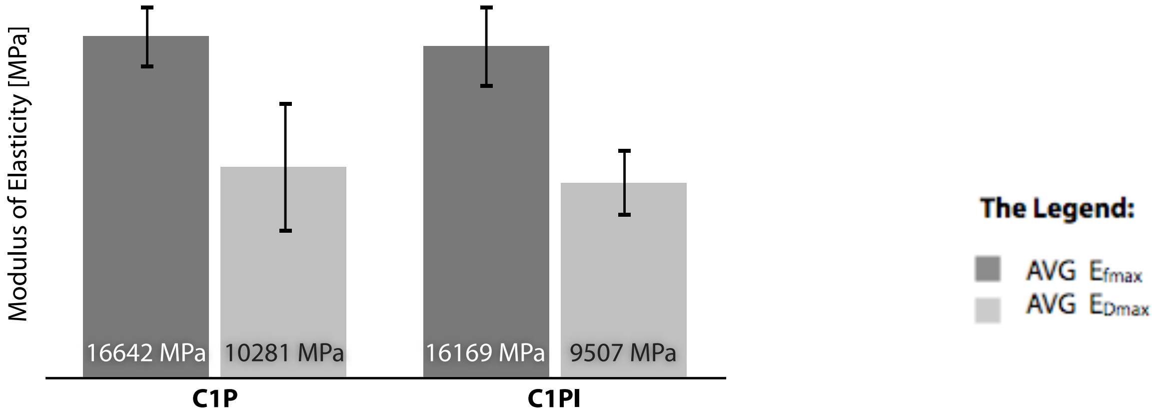

***FIGURE 13**: Influence of the interlayer on stiffness*

3.2.3.4. Influence of different architecture of FRC on stiffness

The impact of the FRC architecture may be evaluated in case the beams of the same volume content of different FRC are compared. It is apparent that the same volume content of unidirectional strip in a layered beam brings larger stiffness compared to same content of fibers in the multidirectional strip due to the more effective orientation of fibres in the strip – see Figure 14. This is in agreement with expectations because the effective modulus of homogenized unidirectional strip is higher than that of multidirectional strip (see the values of modulus at volume content = 1.0 at Figure 14).

The layout of fibers (micro-scale feature) influences the effective stiffness of FRC lamina (meso-scale feature) according to the well-known rules of lamina. On the macro-scale of the layered beam the architecture of fibers is reflected indirectly as one of the factors contributing to the effective stiffness of the FRC layer.

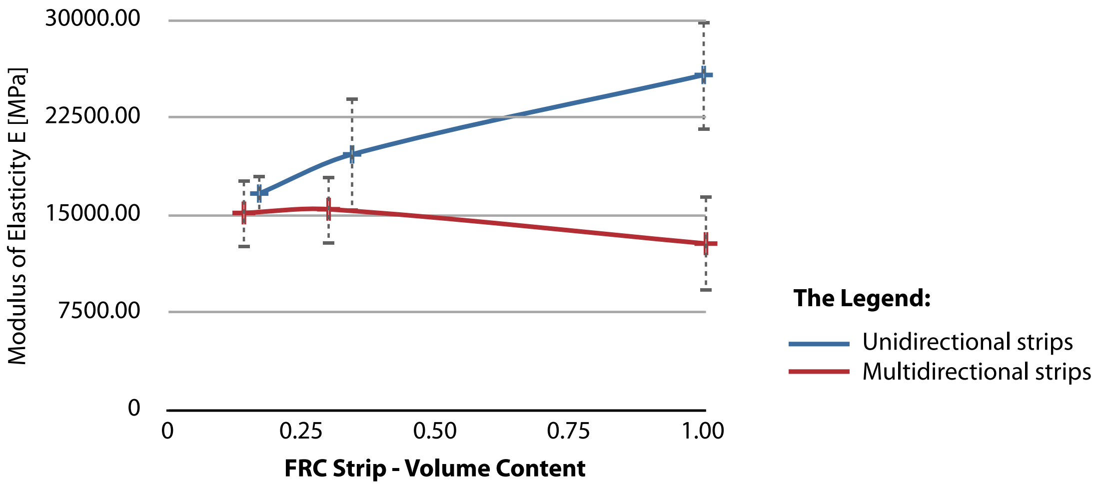

***FIGURE 14**: Influence of FRC architecture on stiffness*

3.2.4 Correlation between experimental data and selected analytical model

The analytical model is based on the Rule of mixture for homogenization of the material in the beam. In a layered composites firm bond between individual layers and uniform strain is expected and consequently the Voigt version of the Rule of mixture was used.

Outputs of the analytical estimation of the effective modulus by the Rule of mixture were compared with experimental data obtained from static three-point tests and DMTA of three point bending as well. Graph in Figure 15 shows the compliance of the values gained in both types of experiments (DMTA and static three point bending) and analytical model.

Based on this comparison it can be stated that Rule of mixture is a robust analytical method to estimate effective resistance to deformation of the layered composites of our interest.

The difference between E_{fmax} and ED_{max} indicates the extension of micro-defects which propagate during the loading and influence the resistance of the specimen to flexure and non-linear elasticity of layered composites as well.

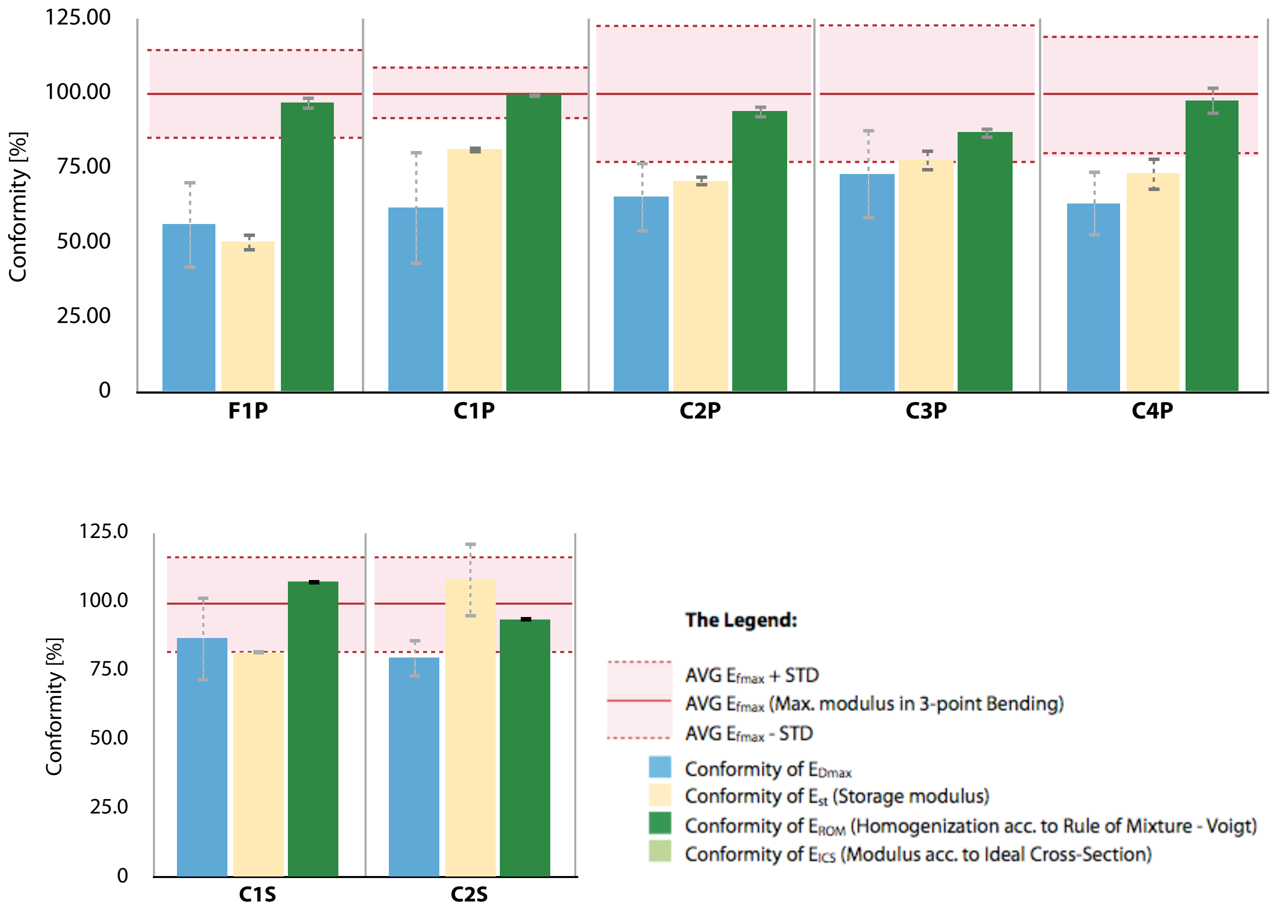

***FIGURE 15**: Comparison of different stiffness moduli*

3.2.5 Maximum stresses and stress distribution

The distribution of the normal stress in bi-material beams follows the scheme on the Figure 16.

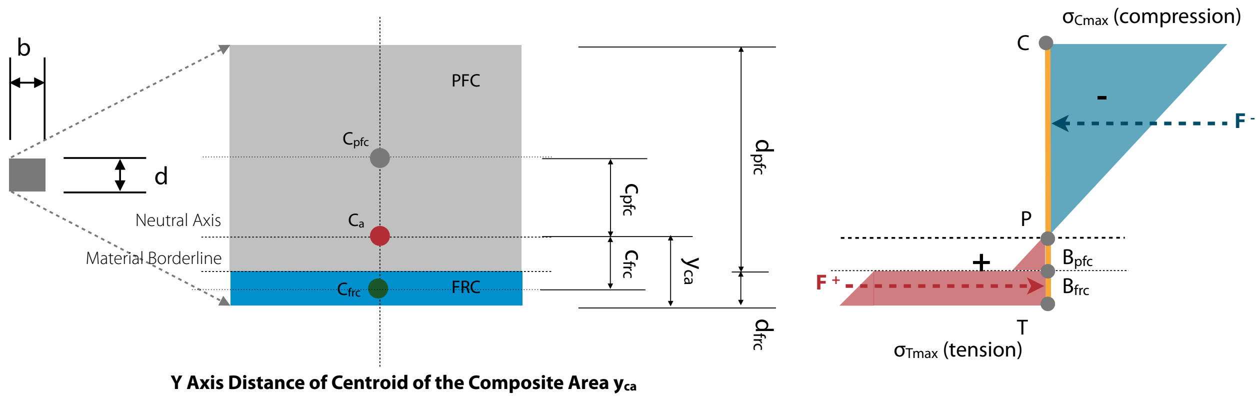

***FIGURE 16**: Normal stress distribution in a bi-layer beam*

Normal stresses (parallel to longitudinal axis x) in the characteristic points of the cross section are as follows:

- Stress σ_T at point T - the farthest point away from neutral axis in the tension part of the cross section
- Stress σ_{BFRC} at point B_{frc} - the point on the material borderline - FRC site
- Stress σ_{BPFC} at point B_{pfc} - the point on the material borderline - PFC site
- Stress σ_C at point C - the farthest point away from neutral axis in the compression part of the cross section
- Longitudinal shear stress $\tau_B = \sigma_{FRC} - \sigma_{PFC}$

3.2.5.1. Influence of the filler content of PFC on normal stress

As shown in Figure 17, there is an influence of filler content of PFC on the longitudinal shear stress τ_B. Larger longitudinal shear between layers exhibits the beam with higher filler content of PFC.

Based on the concept of homogeneization the effective strength of the PFC is a function of the content of a component with higher strength i.e. the filler. It is then apparent that the PFC with higher filler content should have higher strength compared to the PFC with lower filler content. This assumption is in accordance with our experimental results.

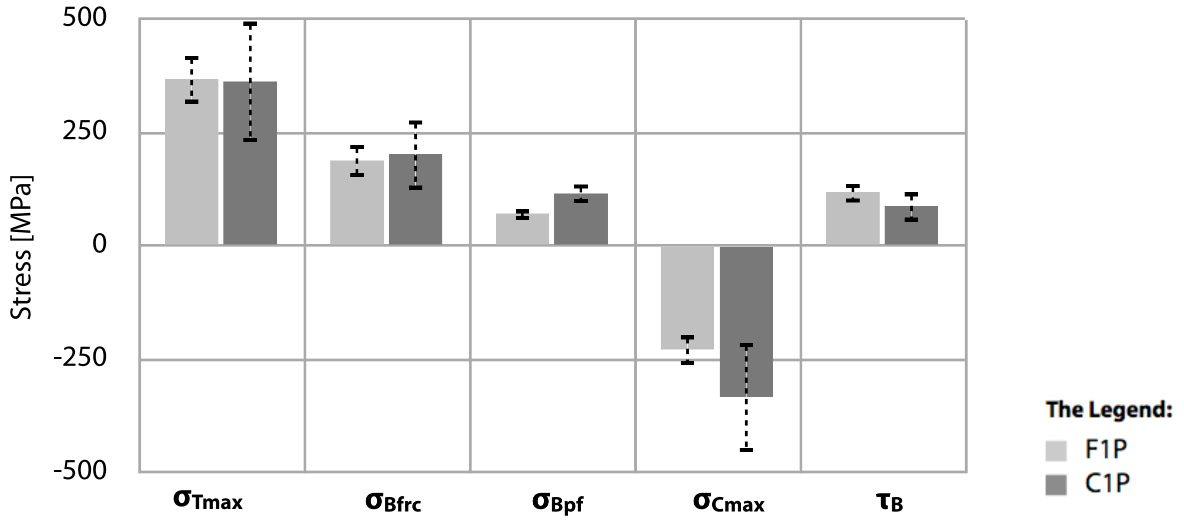

FIGURE 17: Influence of the filler content of PFC on normal stress distribution

The difference between the stresses on the borders of the layer indicates the difference of individual PFCs to carry the load. The normal stresses on the border on the site of the FRC are very close in both specimen (188 ± 36 MPa for F1P and 202 ± 79 for C1P) the respective stresses on the site of PFC are very different (70 ± 14 for F1P and 115 ± 21). This conclusion correlates with different values of maximum compression for both specimens. Larger difference between the strength of adjacent layers creates larger longitudinal shear stress. The relative position of the border of the layer and the neutral axis is the second factor, which influences the value of longitudinal shear on the layers border.

3.2.5.2. Influence of the FRC reinforcement on normal stress

When analysing the influence of the reinforcement on normal stress and its distribution we use the same approach as for stiffness. It is based on the concept of multi-scale modeling where the properties on micro-scale are homogenized and used as an input on meso-scale. Due to the homogenization of FRC layer it is possible to treat FRC as a homogenous layer. In this respect the measure of the reinforcement is the volume content of FRC layer in the specimen measured by the portion of FRC layer on the cross section area of the beam.

The rate of reinforcement has a key impact on the way the stress is distributed across the beam cross section. Figure 18 compares values of respective stresses in individual types of layered beams.

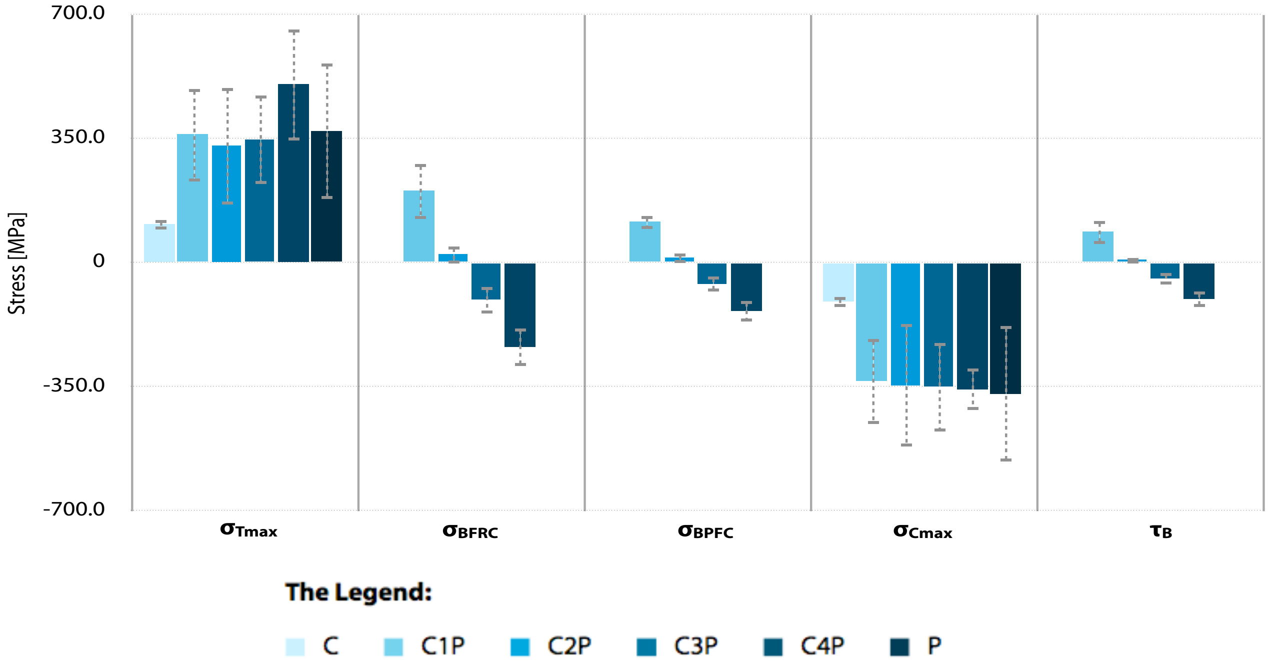

FIGURE 18: *Comparison of the characteristic normal stresses in beams with a different rate of the reinforcement*

Based on the calculated data it is suggested that the volume content of the FRC layer influences normal stress and its distribution in several ways:

- FRC layer increases the maximum stresses compared to PFC mono-layer specimen. It is because FRC has larger strength in tension compared to PFC as indicated in the failure behavior of mono-layer specimen during three point bending tests.
- There was no relation between the volume content of FRC and maximum stresses observed. It is suggested that there is a certain range of effective volume content and out of this range the reinforcing effect of FRC drops down This feature is well known in reinforced concrete.
- The presence of FRC layer and its area in the beam cross section shifts the position of the neutral axis according to the equations of ideal cross section.
- The volume content of FRC layer influences the relative position of the border of the layers and the neutral axis as well. Larger volume content means large area of cross-section occupied by the layer. Providing the FRC layer is placed from the very bottom of the specimen with raising volume content of the FRC the layer’s border goes closer to the neutral axis. With a certain volume content the neutral axis may lay in the area of the cross section occupied by FRC layer .The relative position of the layer’s border and the neutral axis determines the value of longitudinal shear between layers, which influences delamination of the layers.

3.2.5.3. Influence of the interlayer on normal stress

Figure 19 provides a comparison of stresses in layered beams with and without interlayer. It is evident that there is no influence of interlayer on stresses displayed.

This statement does not exclude the hypothesis that the interlayer increases the adhesion between layers. But this eventually increase does not apply to the strength or to stress distribution during the test of given protocol and specimen's geometry. It is in a good agreement with the proposed failure mechanism where delamination of the layer does not activate the final failure in layer beams.

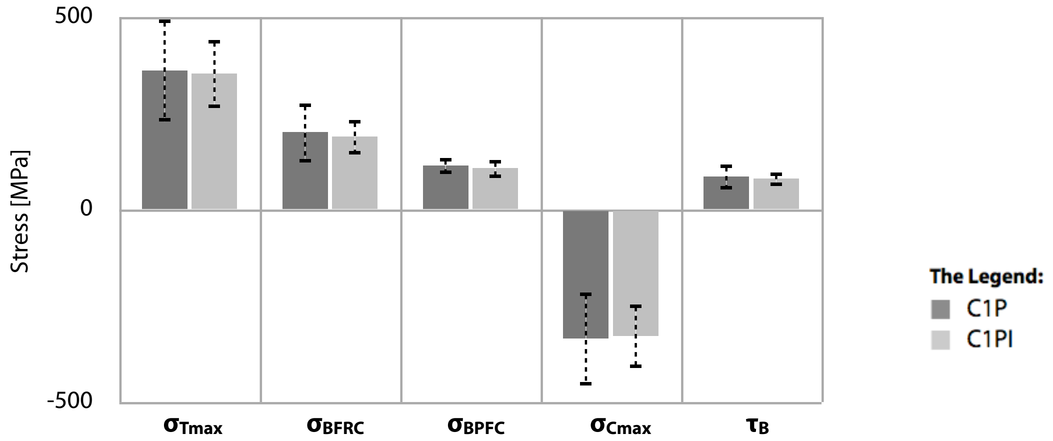

FIGURE 19: *: Influence of interlayer on normal stress*

3.2.5.4. Influence of different architecture of FRC on normal stress

Two types of the beams were chosen in order to study the influence of architecture of FRC on normal stress. Both have neutral axis in about the same position so the influence of different geometry of layers can be neglected. Based on Figure 20 it is evident that the fiber architecture in the layers strongly influences normal stresses in the beam.

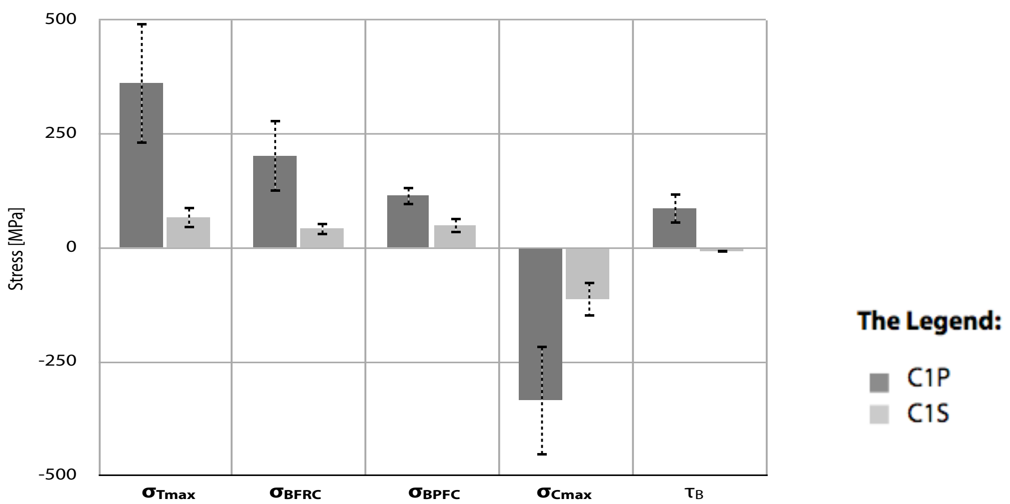

FIGURE 20: *Influence of interlayer on normal stress*

4 CONCLUSIONS

The contents of fibres in FRC ascertained by TGA were in a good agreement with the data supplied by their manufacturers. The content of particles in PFC were in a good agreement with the values typical for low and high viscous dental PFCs. The data from the manufacturer were not available.

The filler content of the PFC layer influences the stiffness and the maximum stresses of layered composites according to the principles of homogeneization of effective properties.

The volume content of the FRC layer (the rate of the reinforcement) influences the stiffness of layered composites. Voigt version of Rule of mixture is in a good agreement with experimental data. Normal stress is influenced by volume content of FRC too. It influences the position of the neutral axis as well as the relative position of the layer's border and the neutral axis. The maximum stress growth with the volume content of the FRC layer in the certain range, out of this range the reinforcing becomes non-effective.

There was no influence of interlayer on overall stiffness nor normal stress and its distribution shown because in the test protocol applied the delamination of the layers does not activate the final failure.

The internal architecture of the FRC layer influences the mechanical response of the layered beams both in stiffness and normal stress. Both effects are in accordance with the concept of effective homogeneization.

Multi-scale modeling is found as a usefull approach to predict and calculate modulus of elasticity and normal stresses of layered composites. Micro-scale is in this respect represented by constituents of PFCs and FRCs, FRC a PFC layer is treated as a meso-scale feature while the whole beam is a macro-scale superstructure.

Following clinical recommendation results from this study:

There is a preferable combination of uni-directional FRC with PFC with higher filler content (CB) and multi-directional FRC and PFC with lower filler content (FLOW).

In this study we focused on how structural features on micro- and meso-scale of constituents influence mechanical response of layered composites used in dentistry. The results confirmed that the internal structure of constituents has an predictable impact on mechanical response.

Layered composites represent a concept in which by using several layouts on different scales new required properties of final superstructure may be obtained.

However, further work is required in order to bring predictable results to dental practice. Clinical testing of different treatment protocols using layered composites would be beneficial in this respect.

5 REFERENCES

[1] Callister Jr., W.D.: Materials Science and Engineering: an introduction. 5th edition, John Wiley and Sons, Inc., 1999

[2] Meyers M., Chawla K.: Mechanical Behavior of Materials, 2nd edition, Cambridge University Press, 2009

[3] Berthelot J.M.: Composite Materials: mechanical behavior and structural analysis, Springer - Verlag, New York, 1999

[4] Kaw A. K.: Mechanics of Composite Materials

[5] Beer R., Baumann M.A., Kielbassa A.M.: Pocket atlas of endodontics, Georg Thieme Verlag, Stuttgart - New York, 2006

[6] Powers J.M., Sakaguchi R.L.: Craig's Restorative Dental Materials. 12th edition, Mosby Elsevier, 2006

[7] Van Noort R.: Introduction to Dental Materials, 3rd edition, Mosby Elsevier, 2007

[8] Kenneth J. Anusavice: Phillip's Science of Dental Materials, W. B. Saunders Company, Philadelphia, 1996

[9] Fouassier J.P., Rabek J.F.: Radiation curing in polymer science and technology, Vol.1: Fundamentals and methods, Eslevier, New York, 1993

[10] Gunduz N.: Synthesis and photopolymerization of novel dimethacrylates, Master of Science Thesis, Virginia Polytechnic Institute and State University, Blacksburg, 1998

[11] Pappas S.P.: Radiation curing, Science and Technology, Plenum Press, New York, 1992

[12] Curtis A.R.: The influence of 'nanocluster' reinforcement on mechanical properties of resin-based composite material, PhD Thesis, University of Birmingham, 2008

[13] Trautmann R.: Morphogenesis of tetrafunctional light cured polymer networks, MSc Thesis, VUT Brno 2002

[14] Cook W.D.: Photopolymerization kinetics of dimethacrylates using the camphorquinone/amine initiation system. Polymer, 1992; 33: 600-609

[15] Venhoven B.A.M.: Dental resin composites - Curing and coherence, PhD. Thesis, University of Amsterdam, 1996

[16] Decker C., Jeckins A.D.: Macromolecules, 18, 1241, 1985

[17] Lanczos C.: The variational principles of mechanics, 4th edition, Dover publications Inc., New York, 1986

[18] Antonucci J.M., et al: Chemistry of silanes: Interfaces in dental polymers and composites, Journal of research of the National institute of standards and technology, vol. 110, Nr.5, 2005

[19] Christensen R.M.: Mechanics of composite materials, Dover Publications Inc., Mineola, New York, 2005

[20] Rösler J., Harders H., Bäker M.: Mechanical behavior of engineering materials: Metals, Ceramics, Polymers and Composites, Springer-Verlag, Berlin, Heidelberg, 2007

[21] de With, G.: Structure, deformation and integrity of materials, Wiley-VCH Verlag GmbH, Weinheim, 2006

[22] Ashby M., Shercliff H., Cebon D.: Materials: Engineering, science, processing and design, Elsevier, 2007

[23] Janssen M., Zuidema J., Wanhill R.J.H.: Fracture mechanics, 2nd edition, Spon Press, 2009

[24]Mishnaevsky L.: Computational mesomechanics of composites, Wiley 2007

[25] Zeman J.: Analysis of composite materials with random microstructure, PhD Thesis, Klokner Institute, Czech Technical University in Prague, 2003

[26] Al Kassem G.: Micromechanical material models for polymer composites through advanced numerical simulation techniques, PhD Thesis, Fakultät für Maschinenwesen der Reinisch-WestfälischenTechnischen Hochshule Aachen, 2009

[27] Weinberger C., Cai W.: Elasticity of microscopic structures - Lecture note, Stanford University, Winter 2004, http://micro.stanford.edu/~caiwei/me340b/content/me340b-lecture02-v03.pdf

[28] Bardella L.: Mechanical behavior of glass-filled epoxy resins: experiments, homogenization methods for syntactic foams, and applications, PhD Thesis, Universita deli Studi di Brescia, Dipartimento di Ingeneria Civile, 2000

[29] Mishnaevsky L., Brøndsted P.: Micromechanical modeling of strength and damage of fiber reinforced composites, Annual report on EU FP6 Project, Risø National Laboratory, Technical University of Denmark, Roskilde, 2007

[30] Suresh S.: Fatigue of materials, Cambridge University Press, 1998

[31] Wong K.J., Yousif B.F., Low K.O., Ng Y., Tan S.L.: Effects of fillers on the fracture behavior of particulate polyester composites, Journal of Strain Analysis, Vol. 45, pp.67 - 78

[32] Cox B., Flanagan G.: Handbook of analytical methods for textile composites, NASA, Langley Research Center, 1997

[33] ISO-Standards. ISO 4049 Polymer-based filling, restorative and luting materials Technical Committee 106 – Dentistry Geneve: International Organization for Standardization. 2000. p. 1-27.

[34] Cowie J.M.G, Arrighi V.: Polymers: Chemistry and physics of modern materials, 3rd. edition, CRC Press, Taylor and Francis, 2008

[35] Chiang Y.: Polymerization Shrinkage with light initiated dental composites, PhD Thesis, Medizinische Fakultät, Ludwig-Maxmilians-Universität, München, 2009

[36] Braga R.R., Ballester R.Y., Ferracane J.L.: Factors involved in the development of polymerization shrinkage stress in resin composites: A systematic review, Dental Materials (2005) 21, 962-970

[37] Van Landuyt K.L. et al., Systematic review of the chemical composition of contemporary dental adhesives, Biomaterials 28 (2007), 3757 - 3785

[38] Ghivari S., Chandak M., Narendra M.: Role of oxygen inhibited layer on shear bond strength of composites, Journal of Conservative Dentistry 2010, Jan - Mar; 13(1): 39-41

[39] Slopek R.,P.: In-situ monitoring of the mechanical properties during the photopolymerization of acrylate resins using particle tracking microrheology, PhD thesis, School of Chemical and Biomolecular Engineering, Georgia Institute of Technology, May 2008

[40] Gauthier M.A., Stangel I., Ellis T.H., Zhu X.X.: Oxygen inhibition in dental resins, Journal of dental research 2005, 84(8), 725 - 729

[41] Rueggeberg F.A., Margeson D.H.: The effect of oxygen inhibition on an unfilled/filled composite system, Journal of dental research 1990, 69, 1652 - 1658

[42] Kučera M.: Vznik makromolekul, VUTIUM Brno, 2003

[43] Decolon C.: Analysis of composite structures, Kogan Page Science, London, 2004

[44] Jančář J.: Mechanics and failure of plastics, VUT Brno, 2005

[45] Kucherov L.: Delamination In Periodically Layered Bi-material Composites, PhD Thesis, Tel Aviv University, 2003

[46] Ding W.: Delamination Anaylsis of composite Laminates, PhD Thesis, University Toronto, 1999

[47] Patton Chan Y.M.: Laminated beam theory based on homogenization, Master Thesis, University of Toronto, 2000

[48] Reddy J.N.: Canonical relationship between bending solutions of classical and shear deformation beam and plate theories, Ann. Solid Structure Mechanics (2010) 1, 9-27, Springer Verlag

[49] Roos R.: Model for interlaminar normal stresses in doubly curved laminates, Dr.sc. Thesis, ETH Zurich, 2008

[50] Tessler A., Di Sciuva M.: Refined zigzag theory for homogenous, laminated composites, and sandwich plates: A homogenous Limit methodology for zigzag function selection, NASA/TP-2010-216214, Langley research center, Hampton, Virginia, 2010

[51] Wanji Ch., Zhen W.: A selective review on recent development of displacement-based laminated plate theories, Recent Patents on Mechanical Engineering, 2008,1, 29-44

[52] Degrieck J, Van Paepegem W.: Fatigue damage modelling of fibre-reinforced composite materials: Review, Applied Mechanics Reviews, 54(4), 2001, 279 – 300

[53] Shah M.B., Ferracane J.L., Kruzic J.J.: R-curve behavior and micromechanisms of fracture in resin based dental restorative composites, Journal of the Mechanical Behavior of Biomedical Materials, Volume 2, Issue 5, October 2009, Pages 502–511

[54] Trautmann R.: Vliv složení na pevnost adheze mezi částicovými a vláknovými kompozity, Dizertační práce, VUT Brno, 2010

[55] Malick P. K.: Fiber reinforced composites, materials, manufacturing and design, 3rd Edition, CRC Press, 2008

[56] ASTM Standard D790 – 99: Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials, 2000

[57] Pardini L.C., Neto F.L., McEnaney B.: Modelling of mechanical properties of CRFC composite under flexure loading, Journal of the Brazilian Society of Mechanical Sciences, Volume 22, Number 2, Campinas 2000

[58] Beckwith S.W.: Composites reinforcement fibers: I – The Glass Family, SAMPE Journal, Volume 45, No. 5, 2009

[59] Mohite N., Kale V., Kumbhar S., Kengar V.: Stress and load-displacemement analysis of fiber composite laminates under different loading conditions, International Journal of Engineering Science & Advanced Technology, Volume 2, Issue 6, 1663-1669, 2012

[60] Pupurs A.: Micro-crack initiation and propagation in fiber reinforced composites, Doctoral thesis, Department of Engineering Sciences and Mathematics, Luleå University of Technology, Luleå, Sweden, 2012

[61] Ferreira R.T.L., Rodrigues H.C., Guedes J.M, Hernandes J.A: Hierarchical optimization of laminated fiber reinforced composites, 10th World Congress on Structural and Multidisciplinary Optimization, May 19-24, 2013, Orlando, FL, USA, *Submitted paper,* 2013

[62] Würkner M., Berger H., Gabbert U.: Numerical study of effective elastic properties of fiber reinforced composites with rhombic cell arrangements and imperfect interface, International Journal of Engineering Science 63, 1-9, Elsevier 2013

[63] Rathnakar G., Shivanan H.K.: Experimental evaluation of strength and stiffness of fibre reinforced composite under flexural loading, International Journal of Engineering and Innovative Technology, Volume 2, Issue 7, 219-222, 2013

[64] Šmilauer V., Hoover Ch., Bažant Z.P., Caner F., Waas A.M., Shahwan K.: Multiscale simulation of fracture of braided composite via repetitive unit cells, Engineering Fracture mechanics, 78 (2011), 901-918

[65] Chen F.: Interface mechanics of layered composite beam-type structures, PhD Thesis, Department of Civil and Environmental Engineering, Washington State University, 2011

[66] Leprince J.G., Palin W.M., Hadis M.A., Devaux J., Leloup G.: Progress in dimethacrylate- based dental composite technology and curing efficiency, Dental Materials, Volume 29, Issue 2, 139-156, 2013

[67] Graciani E.: Numerical Analysis of the single fiber fragmentation test including the effect of interfacial friction, PhD Thesis, Department of Applied Physics and Mechanical Engineering, Luleå University of Technology, Luleå, Sweden, 2010

[68] Pěnčík J.: Dřevěné prefabrikované schodišťové sestavy, Habilitační práce, VUT Brno, *DRAFT*, 2013

ABSTRAKT

Disertační práce se věnuje mechanické odezvě pokročilých vrstevnatých kompozitů pro stomatologické aplikace. Modelová kompozitní struktura je složena z vrstev s částicových a vlákny vyztužených kompozitů. Hlavní důraz byl kladen na zkoumání vlivu strukturálních charakteristik na mikro a mezo úrovni na deformační chování a porušení během statického tříbodového ohybu. Strukturální charakteristiky na mikro a mezo úrovni zahrnovaly orientaci vláken, objemový obsah vláken a částic, adhezi mezi vrstvami a skladbu vrstev. Je prezentován testovací protokol vhodný pro získání spolehlivých dat o vztahu mezi strukturou a vlastnostmi modelových vrstevnatých kompozitů, která jsou relevantní pro klinické aplikace těchto materiálů. Experimentálně zjištěné závislosti tuhosti a parametrů porušení na struktuře jsou podrobeny interpretaci s využitím existujících teoretických modelů. Data získaná z testů DMTA a ze statického tříbodového ohybu byla využita k analýze vlivu strukturálních charakteristik na tuhost a rozdělení napětí v průběhu statické deformace. Na základě těchto analýz jsou navržena základní pravidla pro klinické použití vrstevnatých kompozitů ve stomatologických aplikacích jako jsou minimálně invazivní můstky nebo stabilizační dlahy.

ABSTRACT

The PhD thesis deals with mechanical response of advanced layered composites for dental applications. Model composite superstructure is composed of fiber reinforced and particulate filled composite layers. The main concern was to investigate influence of microscale and mesoscale structural features on deformation behavior and ftracture during static three point bending test. The microscale and mesoscale structural features covered fiber orientation, fiber and particle volume fractions, interlayer adhesion and layering sequence. Testing protocol suitable to obtain reliable experimental data on structure-property relationship for model test specimen relevant for clinical applications is presented. An attempt is made to interpret experimentaly established structure-stiffness and structure-fracture relationships using existing models. Data gained out of static and DMTA three point bending were used to analyze an influence of structural features on stiffness and stress distribution. Based on these analyses, basic rules for clinical applications in dentistry such as minimally invasive bridges and stabilising devices (splints) of layered composites are proposed.