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Uni-axial Test of Particle Filled Dental Composites: Compression and Tension

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Abstract: This paper deals with uni-axial test of three dental composites. As observed in available literature, elastic moduli measured in tension are generally higher than those measured in compression. These findings are confirmed by this study. For all examined materials, the compressive modulus is lower than the tensile one, thus the results do qualitatively well agree with those, obtained from literature. Besides the moduli, other phenomena such as ultimate and yield strength are discussed. Results are compared with data obtained by previous FE simulation. Influences of measurement method as well as certain microstructural phenomena on measured elastic moduli are discussed.

Keywords: Dental Composite; Elastic Modulus; Uniaxial Test

1. Introduction

Particle filled dental composites have become materials of major importance in recent decades. They are used for their superior hardness and aesthetic properties. However, there are significant drawbacks, resulting from their limited longevity. Important problem, investigated by multiple authors [1-4] is the occurrence of microcracks at the tooth/restoration interface. This phenomenon was subjected to FE simulation and several factors of significant effect, such as shape of cavity, technique of curing or a heterogeneous interfacial layer have been discussed. To overcome this difficulty by proper cavity shape, optimization models have been built [5]. All numerical models require an accurate knowledge of mechanical properties of the simulated material. Elastic modulus of a particle filled composite, as one of its most important mechanical properties, can be obtained by several approaches.

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Tensile or compressive uni-axial test, flexural test or nano-indentation are the most cited in available literature [6-8]. Interestingly enough, significant discrepancy between results obtained by particular methods can be observed. In most cases, moduli measured in tension are over 10 GPa, while in compression, measured values are typically between 3 and 5 GPa. Flexural test typically gives moduli are of 6 to 8 GPa, so that they fall between the results of tensile and compressive measurement. Results obtained by nano-indentation are significantly higher, due to the local nature of the method. All aforementioned computational models utilize one value of elastic modulus in both compression and tension. Thus, their results can be questioned, with respect to the experimental findings. Nevertheless, experimental measurements have rarely been carried out in a systematic way, examining one material by several approaches and by one author. This work attempts to present such comparative study and to discuss possible phenomena influencing observed results.

2. Materials

Three commercialy available composites were subjected to the experimental program. Opticor Flow (SpofaDental a.s., Jičín, Czech Republic) consists from a resin (mixture of Bis-GMA, TEGDMA and UDMA) and 77% of a filler (particles of median size 0,7 μm), Filtek Z250 (3M ESPE, St.Paul, MN, USA) based on a resin of Bis-GMA, UDMA a Bis-EMA with 60 % of a glass filler (particles of size 0,1μm to 3,5μm), and Charisma Opal (Heareus Kulzer GmbH, Hanau, Germany) contains 58% of a filler (0,02-2μm) and Bis-GMA resin. Those materials were chosen as typical representatives of the examined class as well as for similarity of their constituents. Typically, elastic modulus of unfilled polymeric dental resins is about 3 GPa, while the most present filler (SiO_2) has a modulus of 70 GPa.

3. Methods

3.1. Tension

3.1.1. Experimental setup

The tensile experiment was carried out using the biaxial testing system Messphysik (Zwick/Roell GmbH, Ulm, Germany) at the Laboratory of cardiovascular biomechanics. Load cells of this system can detect forces of values up to 250N, what is sufficient for the examined specimens (stress-strain curves expected to be linear up to 50 MPa by a cross-section area of 1 mm^2). Elongation of the distance between two marks in the central part of the dog-bone specimen was measured using a video extensometer. Loading rate has been set as 1 mm/min. In order to avoid a possible occurrence of an off-axis load, special fixation system was developed (Fig. 1 and Fig. 2). A pair of cord loops was glued to the specimen and, using yet another pair of loops, it was connected to the machine jaws. This system provides the assurance of a pure tensile load as the cord did not posses any bending stiffness. On the other hand, an imprecise fixation of the cord to the specimen May induce additional bending or torsion load, albeit in far smaller scale than any other fixation device. To successfully utilize such fixation system, attention must be paid to stiffening of knots of the cords, in order to avoid its tightening and sliding during the experiment, causing sudden drops of load. The distinctive longitudinal compliance of the fixation system also does not allow to measure displacement of the loading crosshead, so that the aforementioned video-extensometer must be employed.

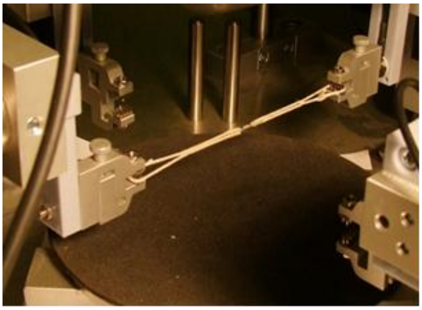

Fig. 1. Specimen for tensile test fixed in testing system

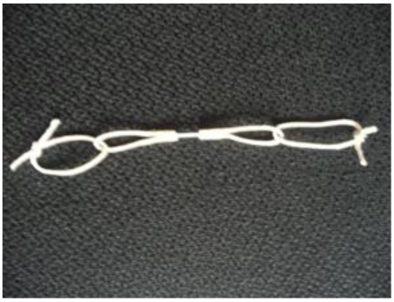

Fig. 2. Specimen for tensile test with the fixation system for avoiding off-axis load

3.1.2. Specimen preparation

The specimens for tensile test are dog-bone shaped (Fig. 3) and their small thickness allows to prepare them in a mold of steel as the used light emitting device secures polymerization of their whole volume and a mold made of a transparent material is not required. The most important dimensions (Fig. 4) of the specimen are width and thickness of the central part as there the elongation is measured. Therefore, these dimensions have been recorded for each specimen and deviation from the nominal dimension has been taken into account by calculation of the stress values. Dimensions of the specimen were selected according to CSN EN ISO 4049 standard, however, they were modified.

Fig. 3. Specimen for tensile test

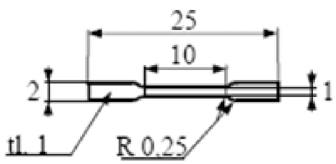

Fig. 4. Dimensions of specimen

To secure the separation of the specimen from the mold, a separator spray (Formula 10, Ambersill Ltd, Bridgewater, Sommerset, UK) was used. In this case (composite/steel), this technique was sufficient and the thin layer, created by the separator did not significantly reduce the dimensions of the specimen. To cure the specimen, LED-based curing lamp (Translux Power BlueTM, Heareus Kulzer GmbH, Hanau, Germany) emitting light of wavelength 440-480 nm and intensity of 1000 mW/cm^2 has been used. In order to cure whole volume of the specimen, curing zones were chosen as mutually overlapping (5x8 mm zones by 25 mm specimen length). Each zone was cured for 20 seconds.

3.2. Compression

3.2.1. Experimental setup

Since the compressive ultimate strength of the examined materials was estimated at over 100 MPa and the cross-section area of specimen is 28.27 mm^2, the experiment was carried out using the testing system Tira 1050 equipped with load cells sensing forces of values up to 20 000 N. Shortening ($\Delta l/l < 0$) of the specimens was measured by recording of the displacement of the loading head. Compression rate has been set at 1 mm/min.

3.2.2. Specimen preparation

The dog-bone shaped specimens utilized for the tensile test are unsuitable for the compressive experiment. In order to avoid stability problems, specimens for compressive testing of this class of materials usually have a cylindrical shape with length/diameter ratio of about 2. Papers dealing with compressive testing of dental composites mention cylinders with dimensions of ø3x6mm [9], ø4x8 mm [10] or ø6x12 mm [11]. Cylindrical specimens of dimensions ø6x12 mm (Fig. 5.a) were chosen for easy manipulation. The specimens were cured in a transparent plexi mold (Fig. 5.b-d), so as to ensure their polymerization in whole volume. The top and bottom face of each specimen was treated with sandpaper with grit size of P800. Nevertheless, dimensions of each individual specimen were recorded and any deviations from the nominal dimension were taken into account by calculation of the stress values.

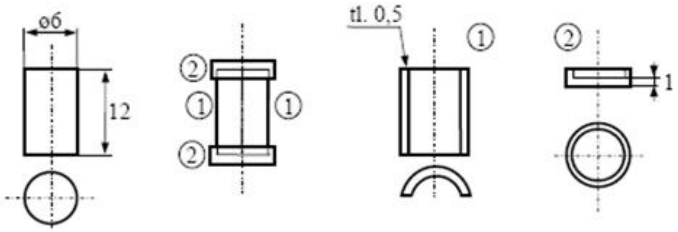

Fig. 5. a. Specimen for compressive test: a. dimensions, b-d. Mold

The separation of the specimen from the mold proved to be a serious problem in the case of composite/plexi-glass interface. The separator spray utilized for the composite/steel interface by the tensile specimens was not sufficient here, so a thin layer of vaseline was applied. This solution was successful but the layer did considerably reduce the dimensions of the specimens which in turn required the more careful measurement of them. Curing of the specimens was done using same equipment as in the tensile case. Curing zones were determined in order to ensure polymerization of whole volume of the specimen. Four mutually overlapping ring-shaped zones were applied (3x8 mm zones by 12 mm specimen’s height) and both the top and base faces were set as one extra curing zone.

4. Results

4.1. Tension

Sets of stress-strain curves were obtained from load-displacement records for each of the materials. The load was applied on the above described fixing system, so the lower regions of the curves can be influenced by processes during its initiation; the linear part of the curve begins typically about 8-10 MPa. Measured stress-strain curves are plotted in figure 6. Value of elastic modulus was obtained as a secant modulus in the linear part of each curve. In the tensile case, the first point of the linear part has been defined by the origin and the end point as the point of 0.2% plastic strain. In case of its absence, the end pint of the curve has been utilized. Thereafter, moduli were averaged for each of examined materials (Table 1). As the

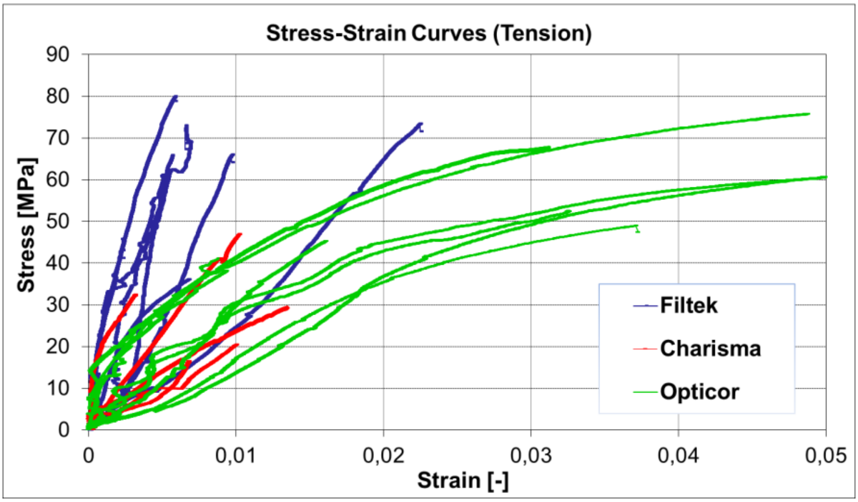

Fig. 6. Tensile stress-strain curves of three examined materials.

output of the camera is provided with significant noise, the stress-strain curves were smoothed for graphical presentation, with the use of an exponential smoothing function. Results of the tensile test provide reliable data by the materials Filtek and Opticor. However, for the results of Filtek, distinctive variance of results must be considered. In the case of Charisma, significant portion of the specimens got broken before recording of the linear part of the curve and was not included in the evaluation. This fact can be ascribed to its brittleness or bubbles in the microstructure. In comparison to the compressive measurement which will be described later, results were obtained for a relatively smaller group of specimens.

4.2. Compression

Analogically to the tensile experiment, elastic modulus was determined as slope of the linear part of the curves. In case of the compressive measurement, the initial part of the curve was affected by roughness of the specimen surface. The linear part has been defined between the point of σ = 20 MPa and the point of 0.2% plastic strain. In case of its absence, the end point of the measured curve defines the second point for secant modulus calculation. Particular moduli were averaged for each of the materials and resulting values were compared with the tensile ones (Table 1).

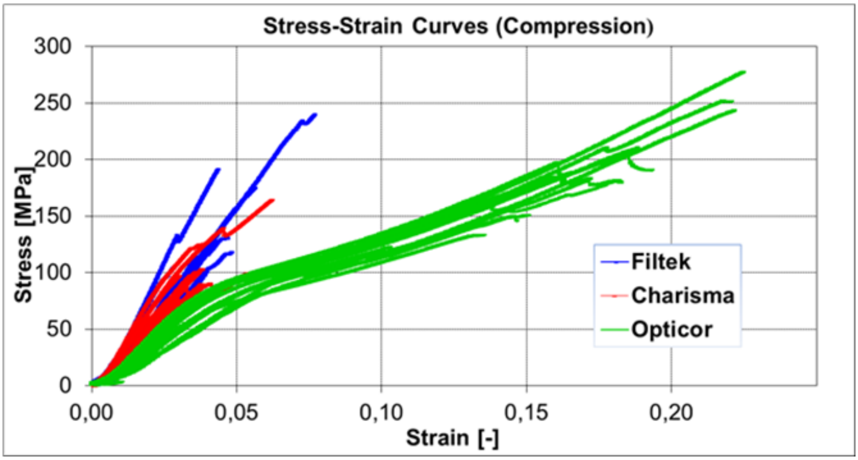

Fig. 7. Compressive stress-strain curves of three examined materials.

For each material, the tensile modulus is higher than compressive one (Table 1). In all cases, except Charisma, the difference is significant and the lowest obtained tensile value is higher than the highest value obtained under compressive test. Ratios obtained for Charisma and Opticor (1.28 and 1.68, respectively), do well meet the results of FE simulation. Despite similar particle content amongst the composites, resulting ratios vary by a factor of more than 3. This can be interpreted as significant dependence on the resins composition, rather than on filler volume fraction. Minor significance of the filler content can be also illustrated by the fact, that the highest-filled material, Opticor Flow (77%), shows the lowest values of elastic moduli in both compression and tension. All samples show distinctive deviation from the arithmetic mean. This can be explained by variety of uncertainty sources. The most significant one, shape, size and distribution of voids in the structure, is of type B (cannot be statistically evaluated), while others belong to the type A (can be evaluated by statistical means). The most important are: uncertainty in force measurement, uncertainty in displacement measurement by crosshead in compressive case and by extensometer in the tensile one and above all the uncertainty in specimen dimensions measurement. The relatively higher deviation of the tensile results can be explained by difference of specimen size. The smaller cross-section of specimen is measured with higher % uncertainty if the same measurement device is used.

Table 1. Elastic Moduli in Tension (E_t) and Compression (E_c): Arith. Mean and Standard Deviation

Material	Elastic modulus in tension [GPa]	Elastic modulus in compression [GPa]	Ratio E_t/E_c
Filtek	13.89 ±4.07 (29.3%)	1.63 ±0.15 (9.2%)	4.13
Charisma	4.07 ± 1.41 (34.6%)	3.17 ±0.52 (16.4%)	1.28
Opticor	2.96 ± 0.61 (20.6%)	3.36 ±0.59 (17.6%)	1.68

5. Discussion

The significant difference between tensile and compressive elastic moduli, observed in each particular case of composite, can be attributed to several factors. The influence of microstructural residual stress on the overall stress-strain response was thoroughly discussed in the work of Prejzek and Mareš [12]. These studies utilize a microstructural FE model to simulate the curing process of the composite and subsequently the tensile and compressive uni-axial experiment. As the material shrinks due to polymerization, stress field occurs in the mictrostructure. Two typical material regions can be distinguished. First, where the value of the stress component in the direction of further load is negative and values of von Mises stress falls distinctively beyond the yield point. In the second typical material region, the values of von Mises stress are below the yield point and the particular stress component is positive. Thus, using any mixing law, lower compressive than tensile elastic modulus must be obtained. Tensile moduli, obtained by an FE simulation of the uni-axial experiment, are higher than the compressive ones by about 60%. Besides the effect of residual stress, minor influence can be ascribed to imperfections of the experimental setups and specimen preparation in particular. The manufacturers refer certain depth of cure for each material, typically 2,5-3 mm. Eichlerová et al. [13] measured decrease of elastic modulus in dependence on the distance from specimen's surface and present E-modulus decreased by 7% in depth of 1 mm. This fact can contribute to the lower elastic modulus measured in compression, since the specimens are significantly thicker in this. However, the difference of 68% observed in case of Charisma and more than three times higher tensile modulus in case of Filtek cannot be explained merely by this phenomenon.

6. Conclusions

Results obtained by systematical uni-axial measurement of three composite materials do well agree with findings of literature review. Particle-filled dental composites have significantly higher elastic moduli in the tensile direction than in the compressive one. Two possible reasons for observed discrepancy have been discussed, namely microstructural plasticity as a major reason and imperfect curing of compressive specimens as a minor one. This work has confirmed a fact traced in literature and should inspire researcher and engineers dealing with dental composites to change their approach to elastic properties of these materials. The elastic modulus should be considered as an asymmetric property, with respect to the direction of load.

Acknowledgements

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References

[1] Braga, R.R. and Ferracane, J.L., "Alternatives in polymerization contraction stress management," *Journal of Applied Oral Sciences*, **15**(3), pp.176-184 (2004). ISSN 1678-7757.

[2] Winkler, M.M., Katona, T.R. and Paydar, N.H., "Finite element stress analysis of three filling techniques for class V light-cured composite restorations," *Journal of Dental Research*, **75**(7), pp.1477–83 (1996). ISSN 1544-0591.

[3] Ensaff, H., O'Doherty, D.M., and Jacobsen, P.H., "The influence of the restoration-tooth in light cured composite restoration: a finite element analysis," *Biomaterials*, **22**(23), pp. 3097-3103 (2001). ISSN 0142-9612.

[4] Fennis, W.M.M., Kuijs, R.H., Barink, M., Kreulen, C.M., Verdonschot, N. and Creugers, N.H.J., "Can internal stresses explain the fracture resistance of cusp-replacing composite restorations?" *European Journal of Oral Sciences*, **113**(5), pp.443-448 (2005). ISSN 1600-0722.

[5] Couegnat, G., Fok, S.L., Cooper, J.E. and Qualtrough, A.J.E., "Structural optimisation of dental restorations using the principle of adaptive growth," *Dental Materials*, **22**(1), pp.3-12 (2006). ISSN 0109-5641.

[6] Van Meerbeek, B., Willems, G., Celis, J.P., Roos, J.R., Braem, M. Lambrechts, P. and Vanherle, G., "Assessment by nano-indentation of the hardness and elasticity of the resin-dentin bonding area," *Journal of Dental Research*, **72**(10), pp.1434-1442 (1993). ISSN 1544-0591.

[7] Zhang, L., Gao, Y., Chen, Q., Tian, M. and Fong, H., "Bis-GMA/TEGDMA dental composites reinforced with nano-scaled single crystals of fibrillar silicate," *Journal of Material Science*, **45**(9), pp.2521–2524 (2010). ISSN 1573-4803.

[8] Masouras, K., Silikos, N. and Watts, D.C., "Correlation of filler content and elastic properties of resin-composites," *Dental Materials*, **24**(7), pp.932–939 (2008). ISSN 0109-5641.

[9] Chen, M.H., Chen, C.R., Hsu, S.H., Sun S.P. and Su W.F., "Low shrinkage light curable nanocomposite for dental restorative material," *Dental Materials*, **22**(8), pp.138-145 (2006). ISSN 0109-5641.

[10] Rafiee, M.A. and Rafiee, J., "Strength properties of light-cured dental restorative composites," in *Proceedings of 35th annual northeast bioengineering conference*, Boston, 2009 (IEEE, Boston, 2009), pp. 31-22. ISBN 978-1-4244-4364-2.

[11] Eliades, G.C. and Caputo, A.A., "The strength of layering technique in visible light-cured composites," *J. of Prosthetic Dentistry*, **61**(1), pp.31-38 (1989). ISSN 0022-3913.

[12] Prejzek, O. and Mareš T., "Particle-filled dental composite: shrinkage induced residual stresses and overall mechanical properties," in *Proceedings of the II international conference on particle-based methods – fundamentals and applications, PARTICLES 2011*, Barcelona, 2011 (CIMNE Universitat Politècnica de Catalunya, Barcelona, 2011), pp.663-672. ISBN 978-84-89925-67-0.

[13] Eichlerová, R., Lukeš, J. and Šepitka, J., "Nanoscale mechanical analysis of dental filling composite and its finite element modelling," *Computer Methods in Biomechanics and Biomedical Engineering*, **14**(sup1), pp.261-262 (2011). ISSN 1476-8259.