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Experimental Stress Analysis

ELASTIC MODULUS OF DENTAL FILLING COMPOSITES: DIFFERENT DETERMINATION METHODS.

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Abstract: Our team-work was focused on dental filling composites investigation with a view to prolong lifetime and minimize probability of failure. The most common failure types were filling fractures, secondary caries and losses of filling. Main reason for failure was stress incidence in filling and adhesive layer causing weakening of tooth-adhesive-composite interface. Stress value could be mathematically analyzed using finite element program. This analysis required knowledge of exact material properties. The goal of work was to determine values of composite elastic modulus by different experimental method: simple tensile test, three-point bending test, nanoindentation test.

1. Introduction

Composite resins and ceramic materials increasingly replaced amalgam and alloy restorative materials in dentistry in the last 20 years. Advantages of composite materials are aesthetic aspects, good biocompatibility, no toxicity and no environmental pollution by mercury waste. Although the use of composite materials in dentistry was successful, there was still a need for improvement. Most common types of dental filling failure were fractures of filling, secondary caries and loos of restoration [1]. Due to mechanisms such as occlusal loading and shrinkage of composites stresses were created in filling and adhesive layer and these stresses caused failures of the tooth-restoration interface and undermine marginal integrity [6]. Other stress contributing factors were cavity shape and unsuitable bonding or composite materials. This problem could be solved by minimizing of tooth-restoration stresses through the development of low-shrinkage and self-bonding dental composites, using of reliable bonding and filling materials or cavity shape optimizing. An ideal cavity shape should minimize stress concentrations along the tooth-restoration interface due to sharp angles or differences in material properties [2]. To optimize the design of cavity a finite element program was used. This analysis required knowledge of exact material properties. Different modern measuring methods allowed determination of material properties of composite and adhesive but also enamel and dentin. The first aim of our work was to determine and to compare results of two destructive methods (simple tensile test, three-point bending test) and two nondestructive methods (nanoindentation and ultrasonic test). Three nowadays perspective materials were chosen for testing: Filtek™ Silorane (3M ESPE, A3), Filtek™ Superme XT (3M ESPE, A2) and Chrisma (Heraeus Kulzer, A2). Results of three-point bending test were presented in this study.

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2. Materials and Methods

2.1. Three-point bending test

Mechanical properties of three different materials (Filtek^{TM} Silorane, Filtek^{TM} Superme XT, Chrisma) were obtained by three-point bending test. Testing was done on 20 rectangular specimens of each material (dimensions: 1mm x 1mm x 12,5mm). Specimens were made from composite paste that was photo-polymerized in a mould by using visible light (Elipar TriLight, λ = 400-515 nm, power density of 800 mW/cm2). Top and bottom of specimen surface was irradiated for appropriate time. Finally all specimens were finished using 360 grit abrasive-coated paper and were stored in distilled water at 37°C for 24hours. The specimen were loaded to a failure at a cross head speed of 0,75 mm/min by a servo-hydraulic testing machine (MTS 858.2 Mini Bionix, CTU in Prague, CZ). The distance between the support beams (diameter 2,4 mm) of the three-point test jig was 10 mm. Flexural modulus E was calculated by the formula

$$E = \frac{F}{d}\frac{l^3}{4bh^3} \tag{1}$$

where l is the distance between the supports (mm), b is the width of the specimen (mm), h is the specimen thickness (mm) and d is the deflection (mm) at load F (N) during the linear region of the load-displacement curve. Resultant values of flexural modulus of each specimen were calculated by median in the linear region of the load-displacement curve. Flexural strength σ was calculated using the equation

$$\sigma = \frac{3Fl}{2bh^2} \tag{2}$$

where F is the peak of the load (N), l is the distance between the supports, b is the width of the specimen (mm) and h is the specimen thickness (mm) [4,5]. The specimens were prepared and tested for each composite in the same way and the mean and standard deviation were calculated.

2.2. Simple tensile test

Preparation and storage of specimens were same as in three-point bending test. Specimens were rectangular and their cross-section area was 1 mm^2 with enlargement on both ends for better gripping into the testing jig. The specimen length was 10 mm between testing jigs. The simple tensile test was performed by a servo-hydraulic testing machine (MTS 858.2 Mini Bionix, CTU in Prague, CZ). The specimens were loaded five-times to one-fifth of maximum load and then to failure. Loading speed was 0.75 mm/min (Figure 1).

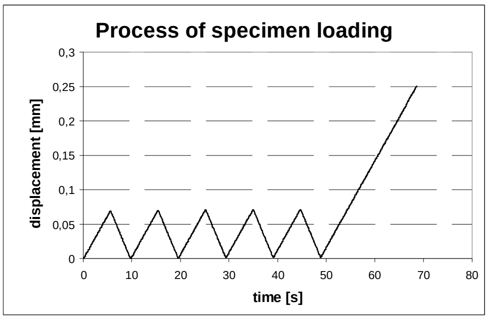

Figure 1: *Time-displacement loading curve of randomly chosen specimen.*

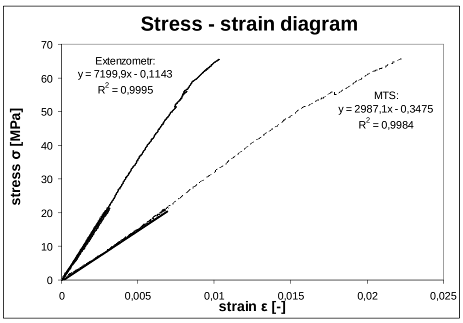

Figure 2: *Difference of stress-strain curves obtained by MTS and extensometer.*

Significant difference was expected between values obtained by extensometer and by MTS. Therefore the use of an extensometer was necessary (Figure 2). Angular coefficient was determined for line-region of the stress-strain curve for each sample. Elastic modulus was calculated as a mean value of these coefficients.

2.3. Nanoindentation test

Specimen for nanoindentation test was a 2 mm high cylinder with a diameter equal to 4 mm. Parallel top and bottom of specimen surface were polished by 2500 grit abrasive-coated paper and by buffing composition. The nanoindentation testing was performed with Hysitron’s TriboLab® at the Faculty of Civil Engineering (CTU in Prague) using the Oliver&Pharr method to calculate reduced modulus E_r [3]. Systems possess the option of in-situ scanning of topography (SPM) and piezo automation with precision of the indent placement less then 1 μm. The elastic modulus E of the sample was determined from the equatin

$$\frac{1}{E_r} = \frac{\left(1-\nu^2\right)}{E} + \frac{\left(1-\nu_{tip}^2\right)}{E_{tip}} \tag{3}$$

where E_r is the reduced modulus measured in an experiment (Figure 3), ν is Poisson’s ratio of the sample, E_{tip} and ν_{tip} are elastic modulus and Poisson’s ratio of the indenter, respectively [3]. The elastic properties of the diamond Berkovich indenter are already known as: E_{tip} = 1141 GPa and $\nu_{tip}=0,07$ [7]. Poisson’s ratio $\nu=0.3$ for resin-composites was used [8].

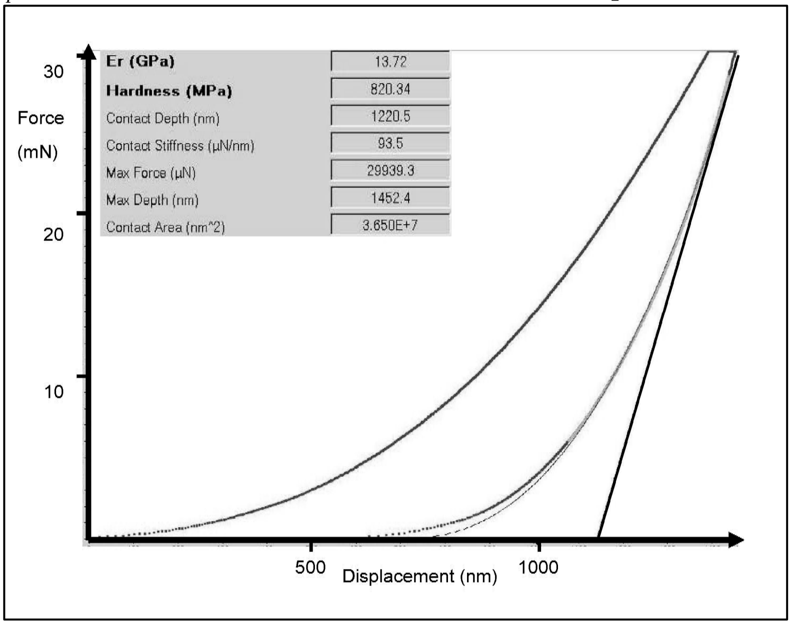

Figure 3: *Load-displacement curve obtained during nanoindentation testing. The depth of the contact circle and slope of the elastic unloading allows specimen modulus and hardness to be calculated.*

3. Results

The values of flexural strength and flexural modulus obtained by three-point bending test are listed in Table 1.

Table 1: Results of three-point bending test: Means of flexural strength and flexural modulus, standard deviations (S.D.), coefficient of variation (CV).

No. of specimen - 20	**Filtek™ Supreme XT**		**Filtek™ Silorane**		**Charisma**	
	flexural strength σ (MPa)	flexural modulus E (MPa)	flexural strength σ (MPa)	flexural modulus E (MPa)	flexural strength σ (MPa)	flexural modulus E (MPa)
Mean	127,52	10013,1	122,62	8256,29	98,58	7483,82
S.D.	21,21	840,22	12,99	895,4	25,82	609,21
CV (%)	16,63	8,39	10,59	10,85	26,19	8,14
Median	129,94	10087,35	123,3	8442,77	100,64	7741,62

The instantaneous values of flexural modulus were dependent on load-displacement curve (Figure 4).

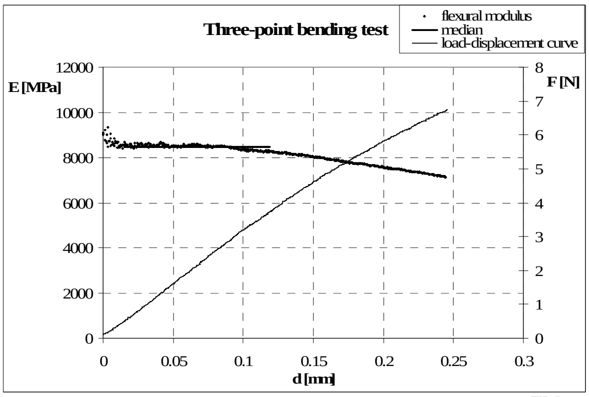

Figure 4: *The time-history of randomly chosen specimen (Filtek™ Silorane). Dots represent calculated instantaneous values of flexural modulus. Thick line is the median of flexural modulus values in the linear region of the load-displacement curve. Thin line represents load-displacement curve of the test.*

4. Discussion

The three-point bending test was performed till specimen fracture. The fracture initiated on tensile side of the specimens and occurred under the applied load in the middle between both lower supports. Load-displacement curve was linear approximately between the deflection of 0,025 mm and 0,15 mm. It means that maximum linear deflection was 15% of specimen thickness. From the results follow that flexural strength and flexural modulus of Filtek™ Supreme XT were higher compared to Filtek™ Silorane and Charisma. There was no significant difference in flexural strength value between Filtek™ Supreme XT and Filtek™ Silorane. However, a significantly higher flexural modulus was observed for Filtek™ Supreme XT. In theory Filtek™ Silorane presented preferable dental restorative composite to Filtek™ Supreme XT and Charisma. Lower shrinkage and modulus together with higher flexural strength of this material could caused lower stress in filling and on margin in adhesive layer of tooth-restoration interface. Thus we can expect better longevity.

5. Conclusion

Failures of dental filling could be minimized by decrease of stress in tooth-restorative interface. The stresses could occur because of shrinkage of dental composites, cavity shape and occlusal loading. One of problem solving was cavity shape optimization analysis done by finite element method program. The aim of work was to obtain values of dental composites material properties for such analysis. The three-point bending test was used to determine flexural strength and modulus. Calculated values of flexural strength σ = 127,5 MPa and modulus E = 10,0 GPa of dental composite Filtek^{TM} Supreme XT were higher in comparison to flexural strength σ = 122,6 MPa and modulus E = 8,3 GPa of dental composite Filtek^{TM} Silorane and flexural strength σ = 98,6 MPa and modulus E = 7,5 GPa of dental composite Charisma.

Acknowledgments

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