

available at www.sciencedirect.comjournal homepage: www.intl.elsevierhealth.com/journals/dema

Fatigue of packable dental composites

Y. Papadogiannis^{a,*}, R.S. Lakes^b, G. Palaghias^a,
M. Helvatjoglu-Antoniades^a, D. Papadogiannis^c

^a Aristotle University of Thessaloniki, School of Dentistry, Department of Operative Dentistry, Thessaloniki, Greece

^b University of Wisconsin, Department of Engineering Physics, Engineering Mechanics Program and Department of Biomedical Engineering, Materials Science Program and Rheology Research Center, Madison, WI, USA

^c National and Kapodistrian University of Athens, School of Dentistry, Department of Biomaterials, Greece

ARTICLE INFO

Article history:

Received 16 September 2005

Accepted 12 January 2006

Keywords:

Composites

Packable

Shear storage modulus

Loss tangent

ABSTRACT

Objective. The purpose of the study was to measure the fatigue properties of four dental resin composites using a dynamic mechanical analysis and to relate the results with viscoelastic properties.

Methods. Dynamic torsional loading was conducted at resonance at 30–50 Hz. Specimens were thoroughly cured and tested dry at 21 °C.

Results. All of the specimens showed a loss of strength following repeated stress, due to material fatigue. The material with the highest shear modulus had the lowest damping and the highest fatigue strength.

Significance. Dental composites exhibit a modest loss of strength due to fatigue. Since mastication involves many cycles of stress during the life of a restoration, fatigue properties should be taken into account in restoration design.

© 2006 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.

1. Introduction

Resin composites were introduced into dental practice as esthetic restorative materials for anterior teeth when they were first developed. However, the growing demand for more esthetic restorations and minimal loss of tooth substance in cavity preparations has made posterior composites an attractive alternative to amalgams [1], and the use of esthetic materials for the restoration of posterior teeth has increased over the past years. This was achieved due to the development of several bonding systems and improved mechanical and physical properties.

The early materials being used as an alternative to amalgam had many problems including marginal leakage because of polymerization shrinkage, low resistance to wear, fracture in the body of the restoration, voids, sensitivity after placement and insufficient proximal contact.

The success rate of these posterior restorations was very high in early clinical evaluations, but started to drop after 5 years. Qvist et al. [2] in Denmark reported that half of Classes I and II restorations were replaced because of secondary caries and bulk fracture of the fillings. In another study in Italian private practices, it was found that bulk and marginal fracture was the reason for 14% of the total replacement of composite restorations [3].

In an effort to increase the performance of composite materials for posterior restorations, the manufacturers increased the filler content and reduced the average filler size to achieve adequate strength and wear resistance to withstand masticatory forces. These composite resins were highly filled and exhibited excellent material properties and clinical performance, but they were difficult to handle and required incremental placement.

* Corresponding author at: 33 Platonos Str, 54 631 Thessaloniki, Greece. Tel.: +30 23 10282878.

E-mail address: papady@dent.auth.gr (Y. Papadogiannis).

0109-5641/\$ – see front matter © 2006 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.

doi:10.1016/j.dental.2006.01.015

Packable composites, sometimes also called condensable composites, have been introduced to the market with high expectations as an alternative to amalgam. They are characterized by a high-filler load and a filler distribution that gives them a different consistency compared to hybrid composites. Packable composites are claimed for use in stress bearing posterior restorations with improved handling properties, as an application technique similar to the manipulation of amalgam can be used for the placement. Easier establishment of physiological interproximal contacts in Class II restorations, the use of metal matrix bands and wooden wedges, and possible bulk curing of the restorations are advantageous. These clinical advantages of packable composite resins captured the interest of clinicians. On the basis of the perceived high-filler load, these materials were expected to exhibit superior physical and mechanical properties besides the improvements in handling.

The changes in composite resins have not been restricted to the filler system. The resin matrix also has an important influence on the properties of composite materials [4,5] and there have also been some modifications in that direction. Multifunctional urethane- and thioether(meth) acrylate alkoxy silanes as sol-gel precursors have been developed for the synthesis of inorganic-organic copolymer ormocer composites as dental restorative materials [6–9]. The alkoxy silyl groups of the silane allow the formation of an inorganic Si–O–Si network by hydrolysis and polycondensation reactions, and the (meth)acrylate groups are available for photochemically induced organic polymerization [7,8]. After the incorporation of filler particles, the ormocer composites can be manipulated by the dentist like a hybrid composite. Ormocers (acronym for organically modified ceramics) are characterized by these novel inorganic-organic copolymers in the formulation, which combines glass-like (inorganic) constituents with polymer (organic) constituents that allow the modification of mechanical parameters in a wide range [6].

Many studies have been undertaken using conventional test methods to investigate the relationship between filler particles in the resin composites and their mechanical properties. Despite all these studies there is still no agreement on the optimum level of filler content for a stress bearing area because the filler size influences the maximum weight percent. High filler loading in composite systems seems to be based on the concept of attaining high mechanical properties as determined by conventional mechanical tests [10]. These tests show that increased filler level results in increased hardness and compressive strength [11–13] and Young's modulus [14,15] as predicted by the composite theory [16,17] while coefficient of thermal expansion [18] water sorption [19], resistance to toothbrush abrasion, and wear by hydroxyapatite, are reduced [12,13].

The effect of filler depends on the type, shape, size and amount used and on the existence of an efficient coupling between filler and matrix resin [20,21]. Many properties change progressively as filler content is increased. For example, compressive strength [11,12,22], hardness [11,12,22,23], flexural strength [22,24] and modulus of elasticity [22,24], all increase when filler volume fraction is increased.

Teeth are subject to many cycles of repeated stress during mastication. Materials subject to such a stress history

undergo material fatigue, which means they fracture at a stress lower than the value required for a single load application. All tissues in the body including bone [25] and dentin [26] undergo repeated stress therefore also fatigue micro damage. Most tissues however, including bone, undergo a continual turnover and remodelling which removes microscopic damage that might give rise to material fatigue. Repeated stress on tooth structure is known to cause micro cracks [27]. Artificial replacements for tissue do not benefit from biological repair processes and therefore are subject to material fatigue damage. Dental materials are in this category. Little has been done to characterize the fatigue characteristics of dental materials. The response to thermal cycling has been looked at [28] in the context of debonding at the interface of inlay restorations and of adhesives [29].

It is the purpose of this study to determine the fatigue response of several dental composites to repeated mechanical shear stress at constant temperature, and to relate the fatigue properties to viscoelasticity of the composites.

2. Materials and methods

Four commercial dental composite filling materials (Alert, Filtek P60, Admira and Synergy as shown in Table 1) that are used in load bearing restorations in posterior teeth were investigated. The composite materials were injected into glass capillary tubes resulting in finished specimens that were 0.85 mm in diameter and the test length was 14 mm for the material Alert and 12 mm for the other three materials. The resins were photo polymerized with a Coltolux 4 light (Coltene Wahaladent, Dentalvertiebs GmbH, Konstanz/Germany) directed at the side of the capillary tube using 40 s exposure time for each 5 mm of length of the tube. Thorough curing was achieved because of the small diameter of the specimens. The specimens were stored in a dry beaker at 21 °C for 24 h after fabrication and subsequently tested dry at room temperature 21 °C.

The specimens were mounted between a rod and a permanent magnet by means of additional composite as a cement, using a jig for centering. The specimens were tested in a torsional creep apparatus (Fig. 1) that has been described in previous articles [30,31].

A permanent samarium cobalt magnet fixed to the end of the specimen generated torque. The magnet produced a torque of 0.0145 Nm/A at the center of a Helmholtz coil. A thin mirror 8.2 mm in diameter and 1.5 mm thick was cemented to the magnet to reflect a laser beam to a chart at a distance D of 944 cm. Tests of quasistatic modulus were done by placing the apparatus under DC current to apply a constant torque. The deformation of the specimen was recorded for a period of time, and then the stress was released to zero. Recovery followed for 10 times the period under load. For fatigue measurements, AC current was used to drive the specimen at torsional resonance. The amplitude of angular displacement of the driven end was measured on the chart at the resonance frequency.

Calculation of stress, modulus, and viscoelastic properties was done as follows. The mirror rotation angle (θ), is given by $\theta = X/2D$, in which X is the chart displacement of the laser beam

Table 1 – Composition of materials

Material	Composition	Manufacturer
Alert packable composite resin	Matrix: functional dimethacrylates of ethoxylated bisphenol-A polycarbonate resins (EBRADMA-PCDMA), crushed glass fibers (CS2). Fillers: Barium-borosilicate glasses, combined with silanated colloid silica (83.5 wt.%, 67 vol.%). Particle size: 0.8 μm. Photoinitiator, amine accelerator, UV absorber, inorganic pigments.	Jeneric/Pentron Inc., 53 North Plains Industrial Road, Wallingford CT 06492
Admira high viscosity ORMOCER	Matrix: anorganic-organic copolymers (ormocers), Bis-GMA, diurethane dimethacrylates, BHT, TEGDMA. Filler: anorganic microfillers 56 vol.% (78 wt.%). Particle size: 0.7 μm	Voco, P.O. Box 767, D-27457. Cuxhaven, Germany
Synergy (compact) packable composite resin	Matrix: BisGMA, BisEMA, TEGDMA resins. Filler: strontium glass, silanized barium glass, silanized amorphous silica, hydrophobed, 59 vol.% (74 wt.%). Particle size: 0.04–2.5 μm (range), Average 0.6 μm.	Coltene Whaledent, Dentalvertiebs GmbH, Fischenzstrasse 39, 78432, Konstanz Germany
Filtec P60 packable composite resin	Matrix: Bis-GMA, UDMA, Bis-EMA resins. Filler: zirconia/silica (83 wt.%, 61 vol.%). Particle size range of 0.01–3.5 μm. Initiators, inorganic pigments.	3M Dental Products, St. Paul, MN, USA

when this displacement is small. For a larger X displacement the angle (θ) is calculated from $\tan \theta = X/2D$.

The weight of the magnet resulted in a constant small axial tensile stress on the specimen. There was no constraint on the specimen for either torsion or extension.

In principle, a torsional load will generate axial deformation in a specimen, however this is a non-linear effect, which is negligible at the small torque levels used. Moreover, the method for measuring torsional angular displacement is totally insensitive to any axial deformation that may occur.

The distribution of shear strain (γ), in a circular cylinder in torsion is

$$\text{strain } \gamma = \frac{r\theta}{L}$$

in this equation, r is the radial distance from the centerline and L is the length of the cylinder. The distribution of the shear stress (σ), depends on the material properties. If it is linearly

elastic or linearly viscoelastic, the shear stress is given by

$$\text{stress } \sigma = \frac{MR}{\pi R^4/2}$$

where R is the specimen radius. Interpretation of torsion results is straightforward when the stress is sufficiently small for the specimen to be linearly viscoelastic. At higher stress, caution is required, since only the outer layers of the specimen experience the peak stresses. Consequently the intrinsic material's non-linearity is underestimated in the results.

2.1. Static shear moduli (G) measurements

For the determination of the shear moduli of elasticity (G) of the tested materials, a constant torque was applied to the specimen for 10 s and then the torque was abruptly released. The angular displacement was recorded and the shear modulus $G = \sigma/\gamma$ was calculated from the equation:

$$G = \frac{2ML}{\pi R^4 \vartheta}$$

The compliance J is the reciprocal of the shear modulus. The shear strain and the shear modulus G at 10 s reflect the instantaneous elastic response of the materials.

2.2. Dynamic experiments-storage shear moduli (G_1) measurements

In a dynamic experiment, when equilibrium is reached and viscoelastic behavior is linear, both stress and strain vary sinusoidally, but strain lags behind the stress. Thus, we write:

$$\text{strain } \gamma = \gamma_0 \sin \omega t$$

$$\text{stress } \sigma = \sigma_0 \sin(\omega t + \phi)$$

where ω is the angular frequency, and ϕ is the phase lag (angle).

The phase angle ϕ between torque and angular displacement may be determined from [32]:

$$\tan \phi = \frac{\tan \delta}{1 - (\omega/\omega_0)}$$

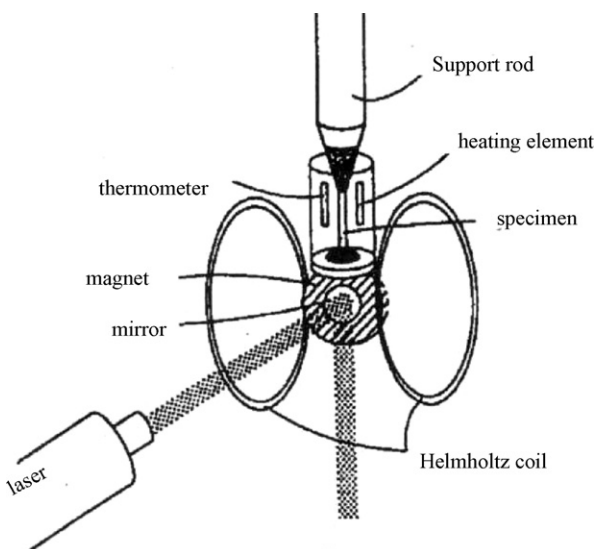


Fig. 1 – Laser based micro-torsion apparatus.

where $\tan \delta$ is the loss tangent and is a measure of damping. It is defined as the ratio of the imaginary part to the real part of the complex modulus G^* . The angle δ is the phase angle between stress and strain sinusoids. $\tan \delta$ is proportional to the energy loss per cycle within the framework of linear viscoelasticity.

The last equation shows that the phase angle ϕ is approximately the same as δ for sufficiently low frequency $\omega \ll \omega_0$, is $\pi/2$ radians at ω_0 , and is π radians for high frequency $\omega \gg \omega_0$. The stress in the maximum strain (resonance case) is: $\sigma = G_1 \gamma_0$, where γ_0 is the maximum strain, because for $\gamma_0: \omega t = \pi/2$.

The storage modulus G_1 (the real part of the complex modulus G^*) is in phase with the strain, whereas, the loss modulus G_2 (the imaginary part of the complex modulus G^*) is 90° out of phase with the strain and is related to dissipation energy. In most cases of stiff solids, G_2 is small compared to G_1 . The complex shear modulus G^* is therefore approximately equal to G_1 and is sometimes loosely referred to as modulus G . G^* is the rigidity of a material and $J = 1/G$ is the compliance of a material. In the dynamic torsional experiment the structural compliance of the material is defined by [32]:

$$\Gamma \equiv \frac{G^* k \theta_0}{M_0}$$

where for a round, straight rod $k = \pi d^4 / 32L$, with d as the rod diameter and L as the rod length, θ_0 is the angular displacement at resonance and M_0 is the torque of the rod upon the magnet.

Viscoelastic parameters were calculated from resonance frequency, ν_0 corresponding to the peak amplitude and the resonance full width $\Delta\nu$, that is the difference between the two frequencies at which the amplitude is half of the maximum. The loss tangent $\tan \delta$ is obtained from the relation [32]:

$$\tan \delta = \left(\frac{1}{\sqrt{3}} \right) \frac{\Delta\nu}{\nu_0}$$

The storage shear modulus (G_1) was calculated from the relation:

$$\nu_0 = \left(\frac{1}{2\pi} \right) \sqrt{\frac{G_1 \pi r^4}{2LI}}$$

where r is the specimen radius, L is its length and I ($4 \times 10^{-7} \text{ kg m}^2$) is the moment of inertia of the magnet calculated from its dimensions and mass. The loss modulus G_2 was calculated from the relation:

$$G_2 = G_1 \tan \delta$$

The dynamic viscosity was obtained from equation:

$$\eta^* = \left(\frac{1}{\omega_0} \right) \sqrt{(G_1 + G_2)}, \quad \text{where } \omega_0 = 2\pi\nu_0.$$

The above simple data reduction is valid for a small loss, i.e. $\tan \delta \ll 1$. The quality factor $Q = (1/\tan \delta)$ is a measure of the sharpness of the resonance curve. A high value of Q correlates with a peaked resonance curve and little damping. Another parameter is the coefficient of decay ($\alpha = \pi \Delta\nu$) which indicates the magnitude of the width between the frequencies at one half the resonance peak of the compliance curve.

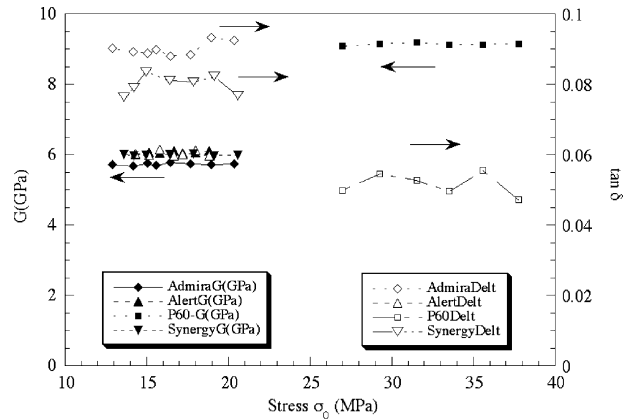


Fig. 2 – Shear modulus G and mechanical damping $\tan \delta$ vs. stress amplitude for the composites studied.

The applied stress was calculated from relation $\sigma = G\gamma$ where G is the storage modulus and γ is the angular deformation. The maximum angular deformation (γ_0) and the maximum (σ_0) where calculated from relations $\gamma = \gamma_0 \sin \omega t$ and $\sigma = \sigma_0 \sin \omega t$ where $\omega t = \pi/2$ in the resonance case.

From each composite material seven specimens were tested, each one under different stress and in the resonance case, until the specimen suffered a fracture. The time to failure was determined using a timer and the number of cycles was calculated as the product of frequency and time.

2.3. Statistical analysis

The means and standard deviations were calculated. The data were analysed statistically using one-way ANOVA and also Tukey's multiple comparison test was used to find any statistical significance between the stress and the number of cycles among the materials investigated.

3. Results

A comparison of shear moduli and mechanical damping values is shown in Table 2 and in Fig. 2. Moduli inferred from static tests and dynamic tests were equivalent within the experimental scatter. This is not surprising since these materials exhibit relatively small mechanical damping, so the change of modulus with frequency in this range will be modest. Filtek P60 had the highest shear modulus and quality factor Q (Fig. 3), the lowest mechanical damping and mean value of coefficient of decay (α) (Fig. 4) among the composites. Fatigue properties are shown as stress to failure versus the log of the number of cycles (Fig. 5). Filtek P60 had the highest strength for any number of cycles. Its torsion endurance limit of about 12 MPa is about twice that of the other composites studied here. These results may be compared with the tensile fatigue strength of adult bovine dentin, about 47 MPa [33]; the corresponding value for shear or torsion would be about a factor of 2.6 lower, or 18 MPa for bovine dentin in shear. Dental bonding adhesive [34] bonded to human enamel was found to have a

Table 2 – Fatigue and viscoelastic properties of examined packable materials

No of specimens	Period, $1/\nu_0$	Resistance to fracture cycles	Loss tangent, $\tan \delta$	Storage modulus, G_1 (GPa)	Loss modulus, G_2 (GPa)	Complex modulus, G^* (GPa)	Dynamic viscosity η^* (Nsm ⁻²)	Shear modulus, G_{static} (GPa)	Quality factor $1/\tan \delta, Q$	Angle, Y (rad)	Stress, σ (MPa)	Log of cycles
Admira												
1	0.02541	3148	0.09243	5.725	0.529	5.749	2.33E+07	5.935	10.82	0.001573	9.007	3.498
2	0.02546	9427.2	0.09333	5.705	0.532	5.730	2.32E+07	5.723	10.71	0.00147	8.386	3.974
3	0.0254	24409.4	0.08843	5.731	0.506	5.753	2.33E+07	5.656	11.31	0.001366	7.830	4.388
4	0.0253	70363.4	0.08792	5.778	0.508	5.800	2.34E+07	5.863	11.37	0.001262	7.294	4.847
5	0.02548	88682.4	0.08975	5.693	0.511	5.716	2.32E+07	5.828	11.14	0.00121	6.891	4.948
6	0.02533	153182.4	0.08891	5.763	0.512	5.786	2.33E+07	5.758	11.25	0.001163	6.705	5.185
7	0.02554	219296	0.0892	5.670	0.505	5.692	2.31E+07	5.972	11.21	0.001106	6.271	5.341
8	0.02544	130880960	0.09025	5.705	0.514	5.728	2.31E+07	5.690	11.08	0.001001	5.713	8.117
Alert												
1	0.02457	4070	0.05958	6.125	0.364	6.136	2.40E+07	6.136	16.78	0.001434	8.781	3.610
2	0.02469	279450	0.0613	6.065	0.371	6.076	2.39E+07	6.076	16.31	0.001379	8.365	5.446
3	0.02475	333704	0.06002	6.035	0.362	6.046	2.38E+07	6.046	16.66	0.001325	7.994	5.523
4	0.02463	41533800	0.06257	6.095	0.381	6.107	2.39E+07	6.107	15.98	0.00127	7.741	7.618
5	0.02457	72232732	0.06242	6.125	0.382	6.137	2.40E+07	6.137	16.02	0.001215	7.444	7.859
6	0.02469	82901880	0.05987	6.065	0.363	6.076	2.39E+07	6.076	16.70	0.00116	7.038	7.919
7	0.02481	90868440	0.06447	6.005	0.387	6.018	2.38E+07	6.078	15.51	0.001106	6.640	7.958
Filtek P60												
1	0.02011	1989.2	0.04714	9.144	0.431	9.155	2.93E+07	9.155	21.22	0.00179	16.369	3.299
2	0.02014	28797	0.04756	9.115	0.434	9.125	2.93E+07	9.125	21.03	0.00169	15.403	4.459
3	0.02012	31808	0.0496	9.133	0.453	9.145	2.93E+07	9.145	20.16	0.001589	14.516	4.503
4	0.02011	1983232.4	0.04841	9.144	0.443	9.155	2.93E+07	9.155	20.66	0.001488	13.611	6.297
5	0.02011	6718166.4	0.04877	9.140	0.446	9.152	2.93E+07	9.152	20.50	0.001387	12.680	6.827
6	0.02017	8678250	0.04983	9.093	0.453	9.104	2.92E+07	9.104	20.07	0.001286	11.691	6.938
7	0.02022	67835510	0.0495	9.041	0.448	9.053	2.91E+07	9.053	20.20	0.001184	10.704	7.831
Synergy												
1	0.02484	60375	0.07674	5.990	0.460	6.008	2.38E+07	6.008	13.03	0.001527	9.146	4.781
2	0.02489	67485.6	0.08221	5.966	0.491	5.987	2.37E+07	5.987	12.16	0.001423	8.490	4.829
3	0.02478	263082	0.0807	6.020	0.486	6.040	2.38E+07	6.040	12.39	0.001319	7.940	5.420
4	0.02483	437440.8	0.08098	5.999	0.486	6.019	2.38E+07	6.019	12.35	0.001214	7.286	5.641
5	0.02486	13469678	0.08354	5.981	0.500	6.002	2.37E+07	6.002	11.97	0.00111	6.638	7.129
6	0.02488	60648317.6	0.07901	5.972	0.472	5.991	2.37E+07	5.991	12.66	0.001057	6.315	7.783
7	0.0248	81814118.4	0.07632	6.011	0.458	6.029	2.38E+07	6.029	13.10	0.001005	6.040	7.913

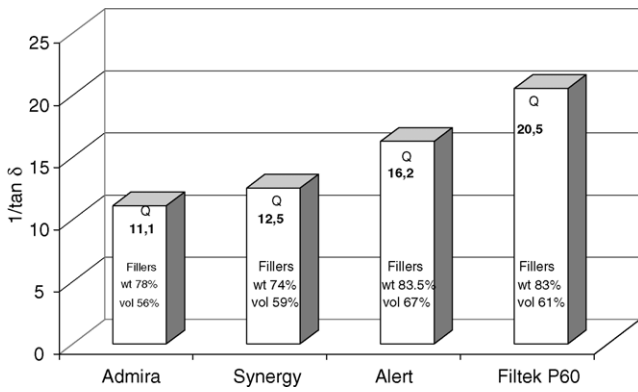


Fig. 3 – Mean value of quality factor Q.

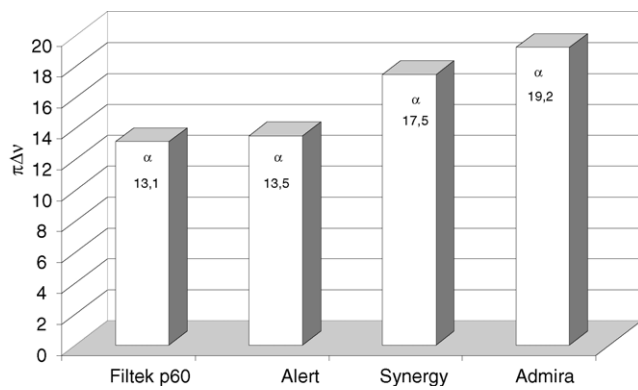


Fig. 4 – Mean value of coefficient of decay (alpha).

static strength of 24MPa and an endurance limit of 10MPa in shear.

4. Discussion

The composite resins can be classified according to the size and amount of the filler particle. Filler morphology is also another important factor that should be investigated. Given

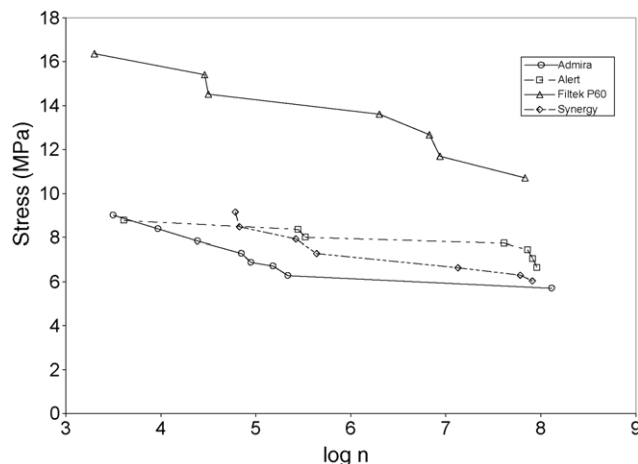


Fig. 5 – Stress amplitude for fatigue fracture vs. log of the number (n) of cycles.

that filler morphology affects the filler loading rate of composites, it may be hypothesized that: (1) composites can be classified by their filler morphology, (2) filler loading is dependent on filler morphology, and (3) filler morphology and filler loading and their silanation, influence the mechanical properties of commercially available composites.

As mentioned before, the properties of composites for example compressive strength [3,5], hardness [3,6], flexural strength [5,7] and modulus of elasticity [5,7] all increase when filler volume fraction is increased and shrinkage naturally decreases as filler volume fraction increases [8].

The results of the work of Iwo Ikejima et al. [35] clearly demonstrated the effects of filler content, filler particle size and filler silanation on mechanical properties. With the increase of filler volume fraction, flexural strength, flexural modulus and shear strength increased up to about 50 vol.% Any further increase in the filler volume above 50% did not cause an increase in the flexural strength and shear strength. There was some evidence that strength begins to decline at very high filler levels (>60 vol.%). However, the modulus of elasticity continued to increase as more filler was incorporated.

In the present study there were four composite resins that belong to the packable category. Alert and Filtek P60 have a filler volume above 60% and belong to the Compact-Filled composites according to the classification of Willems et al. [36]. Synergy and Admira belong to the Midway-Filled composites with filler volume below 60%.

Among the four composites Filtek P60 exhibited the best behavior under fatigue and when compared to the others, its stress to failure versus the log of the number of cycles was the highest, with a statistical significance ($p < 0.001$). This is in accordance with Abe et al. [37] who found Filtek P60 having the highest dynamic elastic modulus among several other materials, including the materials investigated in this study. A material having higher elastic modulus is stiffer and thus can withstand a higher load before deforming. It is important for a restorative material to have an elastic modulus similar to the tissue it replaces. The modulus of elasticity for dentin has been found to have different values by different researchers. It has been reported to be from 13 to 19 GPa [38-40]. The elastic modulus (as a measure of rigidity) has several advantages over other parameters in determining the mechanical properties of the materials. Shear modulus G is usually two to three times less than Young's modulus E as given by the relation for Poisson's ratio $G = E/2[1 + \nu]$ in isotropic materials. The mean value of shear (storage) modulus for P60 is 9.1 GPa and compared to dentin, is satisfactory.

As previously mentioned, Filtek P60 belongs to the Compact-Filled group of composites and because it is more heavily filled than Synergy and Admira it was expected to have better fatigue properties. However, it also showed significantly higher fatigue properties than Alert, which is the material having the highest percentage of fillers. In a previous research [41] that found Filtek P60 having a higher mean of flexural strength than Alert and another material with higher filler concentration, the authors assumed that composites with high filler fractions deteriorate more rapidly. They postulated that highly filled materials present a higher elastic modulus and suffer fragile fracture more easily because they dissipate more energy in the ceramic filler than in the resin binder. Another

reason could be the composition of the fillers, which differs among the materials and are based on zirconia/silica in the case of Filtek P60.

However, in Filtek P60 there is also a difference in the composition of the matrix. The basic component is Bis-GMA, but part of the solvent monomer TEGDMA is replaced by UEDMA and Bis-GMA. It has been shown [42] that the replacement of Bis-GMA and TEGDMA by UEDMA causes an increase in both the tensile and flexural strengths of the matrix. This may be explained by the degree of conversion of the polymer matrix or it can be associated with the ability of the urethane linkage to form hydrogen bonds in the copolymer, which presumably results in restricted sliding of the polymer segments relative to each other. With the correct choice of the content of UEDMA, Bis-GMA and TEGDMA the resin composite may satisfy the needs of use and this may be the case with Filtek P60.

Alert was the composite that had the second highest mean of cycles, however it was not significantly different ($p > 0.05$) from the other two materials that followed (Admira and Synergy). It is the material with the highest filler content (83.5 wt.%, 67 vol.%) and its storage modulus was found to be 6 GPa, a value near to that of dentin. In various studies it has been found to have the highest fracture toughness [43-45], the highest flexural modulus [45,46], Vickers hardness [45,46] and creep resistance [47], but also the highest wear rate [45,46]. In the present study however, Alert did not show better resistance to fatigue than the Midway-Filled composites. The main characteristic of this material is that its filler system contains microfilamentous glass fiber particles 60-80 μm in length and around 10 μm in diameter. According to Adabo et al. [41] the presence of glass filaments may result in a higher elastic modulus with a higher increase in the friability of the material under flexion. Fatigue inside the oral environment is exacerbated by the changes in temperature, chemical water attack and masticatory forces. According to Lohbauer et al. [48] a material with a high initial strength value may not be recommended when focused on fatigue resistance. All materials decrease in strength from fatigue and their ranking can be different when based on fatigue strength compared to the initial strength values calculated, for example, on four point bending tests. This could also happen with Alert, which as previously mentioned [43-47] has been found to have very good fracture mechanical parameters, however, its fatigue properties do not differ significantly from other materials.

Admira is a Midway-Filled composite resin (56% filler volume), which is based on Ormocer technology and exhibits high viscosity. This modification of the matrix however, does not seem to give any advantage over another Midway-Filled composite resin, Synergy, which has the common matrix composition of Bis-GMA and TEGDMA resin. The storage moduli of Admira and Synergy obtained in this study are proportional to the values of Young's modulus E in other studies [37,49] for the same products and they are lower than that of dentin. However, both of these materials did not differ from Alert in their stress versus the log of the number of cycles.

The group of packable composites contains materials with different parameters. Fillers play an important role in determining the properties of the composite resins. However as seen in this study, filler loading is not the only factor that affects the long-term durability of a restoration. The fatigue

strength of the packable composites investigated was not ranked according to the filler volume. The resin matrix, the silanation of fillers and the different types of fillers also play an important role and so the materials that belong to the packable group vary in their fatigue properties. Changes in both the organic and inorganic phases of the composites can alter the properties in order to fulfil clinical requirements. The selection of a material for a posterior restoration should be made only after consideration of all its compositional and mechanical properties like water sorption, wear resistance, modulus of elasticity, hardness and its filler type and size.

REFERENCES

- [1] Fusayama T. Posterior adhesive composite resin: a historic review. *J Prosthet Dent* 1990;64(5):534-8.
- [2] Qvist V, Qvist J, Mjor IA. Placement and longevity of tooth-colored restorations in Denmark. *Acta Odontol Scand* 1990;48(5):305-11.
- [3] Mjor IA, Tofflenetti F. Placement and replacement of resin based composite restoration in Italy. *Oper Dent* 1992;17(3):82-5.
- [4] Kawaguchi M, Fukushima T, Horibe T. Effect of monomer structure on the mechanical properties of light-cured composite resins. *Dent Mater J* 1989;8(1):40-5.
- [5] Peutzfeld A. Resin composites in dentistry: the monomer systems. *Eur J Oral Sci* 1997;105(2):97-116.
- [6] Wolter H, Storch W, Ott H. New inorganic/organic copolymers (ORMOCERs) for dental applications. *Mater Res Soc Symp Proc* 1994;346:143-9.
- [7] Wolter H, Storch W, Ott H. Dental filling materials (posterior composites) based on inorganic/organic copolymers (ORMOCERs) MACRO AKRON (1994);503.
- [8] Manhart J, Hollwich B, Mehl A, Kunzelmann KH, Hickel R. Randqualität von Ormocer- und Kompositfüllungen in Klasse-II-Kavitäten nach Künstlicher Alterung [marginal quality of ormocer and composite restorations in Class II cavities after artificial aging]. *Dtsch Zahnärztl Z* 1999;54:89-95 [in German, abstract in English].
- [9] Hickel R, Dasch W, Janda R, Tyas M, Anusavise K. New direct restorative materials. FDI Commission Project. *Int Dent J* 1998;48(1):3-16.
- [10] Htang A, Ohsawa M, Matsumoto H. Fatigue resistance of composite restorations: effect of filler content. *Dent Mater* 1995;11(1):7-13.
- [11] St. Germain H, Swartz ML, Phillips RW, Moore BK, Roberts TA. Properties of microfilled composite resins as influenced by filler content. *J Dent Res* 1985;64:155-60.
- [12] Li Y, Swartz ML, Phillips RW, Moore BK, Roberts TA. Effect of filler content and size on properties of composites. *J Dent Res* 1985;64:1396-401.
- [13] Chung KH. The relationship between composition and properties of posterior resin composites. *J Dent Res* 1990;69(3):852-6.
- [14] Braem M, Lambrechts P, Van Doren V, Vanherle G. The impact of composite structure on its elastic response. *J Dent Res* 1986;65(5):648-53.
- [15] Braem M, Finger W, Van Doren VE, Lambrechts P, Vanherle G. Mechanical properties and filler fraction of dental composites. *Dent Mater* 1989;5(5):346-8.
- [16] Hashin Z. Analysis of composite materials—a survey. *J Appl Mech* 1983;50:481-505.
- [17] Hashin Z. The elastic moduli of heterogeneous materials. *J Appl Mech* 1962;29:143-50.

- [18] Soderholm KJ. Influence of silane treatment and filler fraction on thermal expansion of composite resins. *J Dent Res* 1984;63:1321-6.
- [19] Oysaed H, Ruyter IE. Water sorption and filler characteristics of composites for use in posterior teeth. *J Dent Res* 1986;65:1315-8.
- [20] Bowen RL. Properties of a silica-reinforced polymer for dental restoration. *J Am Dent Ass* 1963;66:57-64.
- [21] Bowen R. Effect of particle shape and size distribution in a reinforced polymer. *J Am Dent Assoc* 1964;69:481-95.
- [22] Hara R, Nemoto K. Studies on dental restorative composite resin-influence of shape and ratio of filler on the physical properties. *Jpn J Dent Mater* 1986;5:1-16.
- [23] McCabe JF, Wassell RW. Hardness of model dental composites-the effect of filler volume fraction and silanation. *J Mater Sci Mater Med* 1999;10(5):291-4.
- [24] Braem M, Finger W, Van Doren VE, Lambrechts P, Vanherle G. Mechanical properties and filler fraction of dental composites. *Dent Mater* 1989;5(5):346-8.
- [25] Carter DR, Caler WE, Spengler DM, Frankel VH. Uniaxial fatigue of human cortical bone. The influence of tissue physical characteristics. *J Biomech* 1981;14(7):461-70.
- [26] Nalla RK, Imbeni V, Kinney JH, Staninec M, Marshall SJ, Ritchie RO. In vitro fatigue behavior of human dentin with implications for life prediction. *J Biomed Mater Res A* 2003;66(1):10-20.
- [27] Grippo JO. Abfractions: a new classification of hard tissue lesions of teeth. *J Esthet Dent* 1991;3(1):14-9.
- [28] Dietschi D, Moor L. Evaluation of the marginal and internal adaptation of different ceramic and composite inlay systems after an in vitro fatigue test. *J Adhes Dent* 1999;1(1):41-56.
- [29] Frankenberger R, Kramer N, Petschelt A. Fatigue behaviour of different dentin adhesives. *Clin Oral Investig* 1999;3(1):11-7.
- [30] Papadogiannis Y, Boyer DB, Lakes RS. Creep of posterior dental composites. *J Biomed Mater Res* 1985;19(1):85-95.
- [31] Papadogiannis Y, Lakes RS, Petrou-Americanos A, Theodoridou-Pahini S. Temperature dependence of the dynamic viscoelastic behavior of chemical-and light-cured composites. *Dent Mater* 1993;9(2):118-22.
- [32] Lakes RS. *Viscoelastic solids*. Boca Raton, FL: CRC Press; 1998.
- [33] Tonami K, Takahashi H. Effects of aging on tensile fatigue strength of bovine dentin. *Dent Mater J* 1997;16(2):156-69.
- [34] Ruse ND, Shew R, Feduik D. In vitro fatigue testing of a dental bonding system on enamel. *J Biomed Mater Res* 1995;29(3):411-5.
- [35] Ikejima I, Nomoto R, McCabe JF. Shear punch strength and flexural strength of model composites with varying filler volume fraction, particle size and silanation. *Dent Mater* 2003;19(3):206-11.
- [36] Willems G, Lambrechts P, Braem M, Celis JP, Vanherle G. A classification of dental composites. *Dent Mater* 1992;8(5):310-9.
- [37] Abe Y, Lambrechts P, Inoue S, Braem MJA, Takeuchi M, Vanherle G, et al. Dynamic elastic modulus of 'packable' composites. *Dent Mater* 2001;17:520-5.
- [38] Craig GR, Peyton FA. Elastic and mechanical properties of human dentin. *J Dent Res* 1958;37:710-8.
- [39] Watts DC. Elastic moduli and viscoelastic relaxation. *J Dent* 1994;22:154-8.
- [40] Xu HHK, Smith DT, Jahanmir S, Romberg E, Kelly JR, Thompson VP, et al. Indentation damage and mechanical properties of human enamel and dentin. *J Dent Res* 1998;77:472-80.
- [41] Adabo GL, dos Santos Cruz CA, Fonseca RG, Vaz LG. The volumetric fraction of inorganic particles and the flexural strength of composites for posterior teeth. *J Dent* 2003;31(5):353-9.
- [42] Asmussen E, Peutzfeldt A. Influence of UEDMA, BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. *Dent Mater* 1998;14:51-6.
- [43] Knobloch LA, Kerby RE, Seghi R, Berlin JS, Cleland N. Fracture toughness of packable and conventional composite materials. *J Prosthet Dent* 2002;88(3):307-13.
- [44] Kelsey WP, Latta MA, Shaddy RS, Stanislav CM. Physical properties of three packable resin-composite restorative materials. *Oper Dent* 2000;25(4):331-5.
- [45] Manhart J, Kunzelmann KH, Chen HY, Hickel R. Mechanical properties and wear behavior of light-cured packable composite resins. *Dent Mater* 2000;169(1):33-40.
- [46] Manhart J, Kunzelmann KH, Hong Y, Hickel R. Mechanical properties of new composite restorative materials. *J Biomed Mater Res* 2000;53(4):353-61.
- [47] Marghalani HY, Al-Jabab AS. Compressive creep and recovery of light-cured packable composite resins. *Dent Mater* 2004;20(6):600-10.
- [48] Lohbauer U, von der Horst T, Frankenberger R, Krämer N, Petschelt A. Flexural fatigue behavior of resin composite dental restoratives. *Dent Mater* 2003;19:435-40.
- [49] Abe Y, Braem MJA, Lambrechts P, Inoue S, Takeuchi M, van Meerbeek B. Fatigue behavior of packable composites. *Biomaterials* 2005;(26):3405-9.