Clinical performance of resin composite restorations: the value of accelerated in-vitro testing
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CHAPTER 4

Fatigue behavior of dental resin composites: Flexural fatigue

\textit{in vitro} vs. six years \textit{in vivo}
Chapter 4

Introduction
During the last decades, resin-based composite materials were optimized with a special focus on amalgam replacement. Thus, resin composites were more and more improved for application even in occlusally stressed areas. In order to simulate clinical conditions in the laboratory, it is important to observe fatigue phenomena as an important factor during biodegradation processes of dental biomaterials.

In former times that have been characterized by less effective adhesive technology and higher polymerization stresses generated by older resin composite materials, gap formation and recurrent decay were the predominant reason for failures of bonded resin composite restorations. Today, this is still true, but fatigue fractures after several years under clinical load gain importance as reason for failure.

Fatigue in dental restoratives is influenced by corrosive water attack at 37°C and by subcritical cyclic masticatory forces having been estimated to be 5-20 MPa. Contemporary approaches to fatigue principles describe fracture processes in three stages: crack initiation, slow crack growth, fast fracture. Especially crack initiation time and slow crack growth time are determining fatigue resistance of an individual material. Crack initiation originates from surface and subsurface microcracks, porosities, or filler particles. Cyclic loading is able to drive a crack, and additional water exposure causes a variety of further weakening effects on resin composite materials, i.e. degradation of the filler–matrix interface, swelling, or visco-elastic effects on the matrix which all are able to accelerate slow crack growth.

Today it is still not fully understood whether and to what extent we are able to predict clinical behavior of dental biomaterials such as resin composites based on in vitro research. Therefore, the aim of this study was to compare fatigue behavior of two different resin composites using an array of laboratory parameters (Young's modulus, flexural strength, flexural fatigue limit) and analyze resin composite restorations in the course of a randomized prospective clinical long-term trial over 6 years. This is the maximum approach to thoroughly evaluate dental materials simultaneously in vitro and in vivo.
Materials and Methods

Grandio

Grandio (shade A3, Voco, Germany), a nanofilled hybrid resin composite, was used in this study. The resin matrix consisted of bisphenol glycidyl methacrylate (BisGMA) and triethylene glycol dimethacrylate (TEGDMA), mixed in a ratio of 3:1. Further 71.4 vol% (87 wt%) inorganic fillers were compounded, being 30% nanosized fillers. Camphoroquinone served as photoinitiator. Grandio is clinically indicated as a universal composite for anterior and posterior restorations.

Tetric Ceram

The light-curing dental restorative Tetric Ceram (shade A3, Ivoclar Vivadent, Liechtenstein) was used in this study. The material was based on a bisphenol glycidyl methacrylate (BisGMA)/urethane dimethacrylate (UDMA)/triethylene glycol dimethacrylate (TEGDMA) resin matrix, with camphoroquinone as photoinitiator and 60 vol% (78 wt%) inorganic filler content. Tetric Ceram is clinically indicated as a universal fine particle hybrid resin composite for anterior and posterior restorations.

Specimen preparation

Rectangular specimens with the dimension 2 x 2 x 25 mm were produced using a metal/glass mold and light-curing on five overlapping spots of 8 mm diameter. The upper and lower side of the bar were cured with a commercial halogen light curing unit (Elipar Trilight (750 mW/cm²), 3M Espe, Germany). The illumination time on a single spot was 40s. The procedure followed the manufacturers’ recommendation and ISO 4049 standard. The specimen flanges were ground under an angle of 45° using SiC paper (1200 grit). All specimens were stored for 14 days in distilled water at 37°C, deviating from the ISO 4049 standard.

Elastic Modulus (EM)

To determine the elastic response of the composites, the Young’s moduli were calculated from load-displacement curves in a four-point bending test setup. The specimens (n=10) were positioned in the test rig (distance between the lower supports: 20mm, upper fins: 10mm) of a universal testing machine (Zwick Z2.5, Zwick, Ulm, Germany) and loaded up to 20 MPa with a crosshead speed of 0.75 mm/minute. By the linear-elastic relationship between stress (in the range between 10 to 20 MPa) and
strain the EM was calculated. An extensometer served to measure the true deflection (system compliance) directly at the lower side of the specimen.

Fracture strength (FS)

To evaluate the initial flexural strength (FS), the four-point bending test was used (n = 15). Fifteen bars of each material were brought into the four-point test rig and loaded until fracture with a crosshead speed of 0.75 mm/minute in a universal testing machine (Z 2.5, Zwick, Germany). The measurements were carried out in distilled water at 37°C.

Flexural Fatigue Limit (FFL)

The flexural fatigue limits (FFL) of the composite materials were determined for 10^5 cycles under equivalent test conditions at a frequency of 0.5 Hz (n=25). The “staircase” approach was used for fatigue evaluation. For every cycle the stress alternated between 1 MPa and the maximum stress. Tests were conducted sequentially, with the maximum applied stress in each succeeding test being increased or decreased by a fixed increment (5 MPa) of stress, according to whether the previous test resulted in failure or not. The first specimen was tested at approximately 50% of the initial flexural strength value. As the data are cumulated around the mean stress, the number of specimens required is less than with other methods (n=25). The test stopped at a certain level below which no further failure occurred.

The mean flexural fatigue limit (FFL) was determined using Eq. 1 and standard deviation, using Eq. 2, respectively:

\[
FFL = X_0 + d \left( \frac{\sum n_i}{\sum n_i} \pm 0.5 \right)
\]

(1)

\[
SD = 1.62d \left( \frac{\sum n_i \sum i^2 n_i - (\sum in_i)^2}{(\sum n_i)^2} + 0.029 \right)
\]

(2)

where \(X_0\) is the lowest stress level considered in the analysis and \(d\) is the fixed stress increment. To determine the FFL, the analysis of the data was based on the least frequent event (failures versus non failures). In Eq. 1 a negative sign was used when
the analysis was based on failures. The lowest stress level considered was designated as \( i = 0 \), the next as \( i = 1 \), and so on, and \( n_i \) was the number of failures or non-failures at the given stress level.\(^{6,13,17}\)

**Statistical treatment**

The strength data (FS and FFL) were statistically treated using a one-way ANOVA test followed by a modified LSD post-hoc routine (\( p < 0.05 \)).

**Clinical study**

In the course of a randomized clinical study with approval of a local ethics committee (University of Erlangen, Germany), 30 subjects (23 female, 7 male, mean age 32.9 (24-59) years) with a minimum of two restorations to be placed in different quadrants received at least two different Class II restorations (52 MO/OD, 16 MOD or more surfaces, no cusp replacements) in a random decision.\(^{26}\) Thirty six Grandio restorations were bonded with Solobond M (Voco) and 32 Tetric Ceram restorations were bonded with Syntac (Ivoclar Vivadent). The cavities were cut using coarse diamond burs under profuse water cooling (80 µm diamond, Komet, Lemgo, Germany), and finished with a 25 µm finishing diamond. Inner angles of the cavities were rounded and the margins were not bevelled. After cleaning and drying under rubber dam isolation (Coltene/Whaledent Inc., Altstätten, Switzerland), adhesive procedures were performed with Solobond M (2-step etch-and-rinse adhesive) and Syntac (4-step etch-and-rinse adhesive). The resin composite materials were applied into the cavity in layers of approximately 2 mm thickness and adapted to the cavity walls with a plugger. Each layer was light cured for 40 s (Elipar Trilight, 3M Espe, Seefeld, Germany). The occlusal region was sculpted as exactly as possible under intraoral conditions, avoiding visible overhangs. The light-emission window was placed as close as possible to the cavity margins. The intensity of the light was checked periodically with a radiometer (Demetron Research Corp., Danburg, CT, USA) and was found to be consistently above 650 mW/cm\(^2\). Visible overhangs were removed with a scaler and the rubber dam was removed. Contacts in centric and eccentric occlusion were checked with foils (Roeko, Langenau, Germany) and adjusted with finishing diamonds (Komet Dental, Lemgo, Germany), shaped with flexible discs (3M Dental, St. Paul, USA), super-fine discs (3M Dental, St. Paul, USA) and polishing brushes (Hawe-Neos Dental, Bioggio, Switzerland). A fluoride varnish (Elmex Fluid, GABA, Lörrach, Germany) was applied with a foam pellet (Pele-Tim, Voco, Cuxhaven, Germany) to complete the treatment.
Impressions (Dimension Penta and Garant, 3M ESPE, Seefeld, Germany) were taken at baseline, after 4 and 6 years and were used to make epoxy replicas (Alpha Die, Schütz Dental, Germany) for x30 magnification cast analysis under a scanning electron microscope (SEM) evaluating fatigue characteristics such as voids, chippings or cracks within the resin composite, and 22 of them (n=11 per group) were subjected to SEM margin analysis at x200. The replicas with the longest evaluable margins were selected randomly. All in vitro and in vivo replicas were mounted on aluminum stubs, sputter-coated with gold and examined under a SEM (Leitz ISI 50, Akashi, Tokyo, Japan) at x200 magnification. SEM examination was performed by one operator having experience with quantitative margin analysis who was blinded to the restorative procedures. The marginal quality between resin composite and enamel was expressed as percentage of the entire judgeable margin length, i.e. margins having been accessible by the impression material in vivo. Marginal qualities were classified according to the criteria “continuous/gap-free margin” and “gap/irregularity” for another study, here the focus was set on marginal breakdown and cracks. Non-judgeable parts and artifacts were excluded. Afterwards the percentage of “marginal fracture areas” in relation to the individual judgeable margin was calculated as marginal fracture score. For better comparison with in vitro results, gap-free margins were scored whether there was a negative step or not.

Statistical analysis was performed using SPSS 14.0 (SPSS Inc., Chicago, IL, USA). As the majority of groups in each of the two investigations did not exhibit normal data distribution (Kolmogorov-Smirnov test), non-parametric tests were used (Wilcoxon matched-pairs signed-ranks test, Mann-Whitney-U test) for pairwise comparisons at the 95% significance level.

Results
Results of the laboratory part are displayed in Table 1. It was shown that Grandio exhibited a significantly higher elastic modulus (p<0.05) and flexural fatigue limit (p<0.05). Initial fracture strengths were not significantly different (p>0.05). Results of the clinical investigation are shown in Tables 3 and 4. Irrespective of the resin composite used, significant changes over time were found for all criteria applied in clinical examinations (Friedman test; p<0.05). The main reason for the degradation of the occlusal surface of the restorations was an increased surface roughness (41%
after 4 and 27% after 6 years) and more chipping especially in the proximal ridge area (36% after 4 and 35% after 6 years; Fig. 2). Voids were obvious mainly after 6 years (clinically 44% and SEM 73%; Table 3 and 4). Except for the criterion “wear” after 6 years (Pearson correlation coefficient 0.671) no correlation was calculated between the clinical and cast evaluation (p>0.05; two-tailed correlation). Neither restorative materials nor localization of the restorations (upper or lower jaw) had a significant influence on the surface degradation at baseline and after 4/6 years (p>0.05; Mann-Whitney U-test). However, molar restorations performed worse than premolar restorations regarding the clinical criterion “wear” after both 4 and 6 years (premolars: 22% bravo after 4 and 44% after 6 years vs. molars: 61% bravo after 4 and 69% after 6 years).

Criteria and results of the x30 magnification cast analysis under the SEM are displayed in Table 4. Here also, no significant differences were computed between the materials Grandio and Tetric Ceram. Only in the x200 SEM analysis of restoration margins, marginal breakdown areas were more often recorded for Tetric Ceram (7.9%) vs. Grandio (4.8%). Examples for clinical images and corresponding SEM pictures are explained in Figures 1-5. Predominant findings in both species of investigations were slightly exposed restoration margins after 6 years of clinical service due to mainly wear in the contact free areas (CFA; Figures 1b, 2b, 3b, 4b) and more roughened surfaces in the occlusal contact areas (OCA).

**Discussion**

Biomaterials labs daily try to simulate clinical conditions in order to predict clinical behavior of dental biomaterials. This is the only and most important justification of the existence of this kind of laboratory. Without clinical relevance, what should be the reason for in vitro research?

Beside other co-factors for clinical success with resin-based composites such as marginal integrity, wear, biocompatibility and absence of postoperative hypersensitivities, fatigue is a major factor during biodegradation of restorative materials. Fatigue loading has gained importance in materials science not only regarding adhesion, since even for restorative materials the initially high flexural strength values suffered a reduction of 50% after fatigue loading.
The chosen in vitro setup of the present study was shown to give reliable and reasonable results in several studies with an array of different classes of materials. On first sight it may be awkward to evaluate a restorative material as simple beam in a 4-point bending device, since the same material is always adhesively bonded under clinical conditions. However, results from previous studies as well as the present results demonstrate that FFL analysis of dental biomaterials gives important results for their intraoral use in stress-bearing restorations. Facing the present results in both aspects in vitro and in vivo, it is crucial to achieve equal or even similar test conditions in both setups.

One fundamental point here is light polymerization. On one hand, we had to stick to previously published curing protocols to get comparable results to former investigations, on the other hand, light-curing under clinical conditions should at least be achievable. So it might be a consequence that in vitro specimens are still more intensively light-cured than the same materials having been applied in vivo. The same is true for storage times in vitro, so we used the well-established protocol of 2 weeks water storage whereas loading began immediately intraorally.

Another critical point in the present simulation of clinical circumstances is the in vitro setup in general. In vitro we used simple beams having been mechanically loaded as bars and not as bonded resin composite restorations being stabilized inside the tooth. Here, thermomechanical loading may be much closer to the real clinical situation, however, also in a chewing simulation chamber with more complex and individually styled restorations in natural teeth forces are more complex to handle.

The normal way of testing dental biomaterials is to conduct preclinical issues first and then carry out clinical trials. However, when in vitro and in vivo research is carried out simultaneously as in the present paper, an appropriate comparison of both ways of investigations is possible. So since 1998, we decided to perform in vitro and in vivo research with the same materials simultaneously with both resin composites and ceramic materials. This may be the most appropriate way to explain what happened clinically and why it happened as well. Furthermore it is easier to determine predictable parameters in the laboratory.

Compared to other classes of restorative materials such as glass ionomer cements, both resin composites under investigation provided sufficient initial flexural strengths
of >100 MPa. However, both Young’s modulus and flexural fatigue limit were significantly higher for Grandio. In the present study, FFL of investigated materials was 63 MPa for Grandio and 44 MPa for Tetric Ceram. These findings were not statistically different at the initial stage of flexural strength. After mechanical fatigue, the FFL of Grandio was 55% of the initial flexural strength, whereas Tetric Ceram dropped to 45% of its initial flexural strength values. Regarding both aspects, physicochemical characteristics were slightly better for Grandio which also exhibited a somewhat increased filler load of 87%wt vs. 78%wt in Tetric Ceram. Facing the overall clinical performance of both materials, slightly superior materials characteristics for Grandio did not result in a significantly better clinical outcome, at least after 6 years of clinical service. Only when dental restorative materials have flexural fatigue limits of less than 20 MPa, it seems to be critical for clinical survival with more frequent catastrophic bulk fractures even after considerably shorter clinical observation periods. In a previous study under identical test conditions in vitro as well as in vivo, Solitaire provided a FFL of 18 MPa which lead to unacceptably high marginal and bulk fracture rates in vivo in the course of a prospective clinical study.

Finally, the significantly higher FFL for Grandio when compared to Tetric Ceram led to clinical consequences having been detected under the SEM during the marginal quality evaluation: Grandio restorations exhibited significantly less marginal breakdown (4.8%) than Tetric Ceram restorations (7.9%). Although Grandio restorations showed inferior marginal quality in a previous clinical trial, they performed slightly better according to marginal fatigue characteristics in the present investigation. However, these are microscopic findings suffering limited clinical impact for the present observation period of 6 years when it comes to clinical survival as the major criterion.

Up to now, wear was the predominantly detected degradation factor over time. In contrast, the influence of the clinical operator is still a major factor for clinical success, maybe more than the difference between an FFL of 40 MPa vs. 50 MPa.
References


