Flexural Fatigue Behavior of Machinable and
Light-activated Hybrid Composites for Esthetic Restorations

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Running title: Flexural fatigue strengths of esthetic composites

Abstract: The purpose of this study was to determine the flexural strength and flexural fatigue strength of a machinable composite (GN-I) and three hybrid composites (Artglass, Estenia, and Gradia). Specimens ($2 \times 2 \times 25$ mm) were polymerized in a laboratory-photo-curing unit and then immersed in water at 37°C for 24 h, 6 months, and 1 year. After each immersion period, flexural strengths (4-PFS) were measured at a cross-head speed of 1 mm/min in 4-point bending (span = 20 mm; distance between inside loading points = 10 mm). The cyclic fatigue test was performed at 2.0 Hz on a fatigue tester. The staircase method was employed for flexural fatigue strength (FFS) using a 5,000 cyclic load limitation, 5 MPa stress increment, and 20 specimens for each material. Both 4-PFS and FFS of the machinable composite after all immersion periods were significantly greater than those of the three hybrid composites. The FFS results followed the same pattern as those of the 4-PFS. The Estenia material exhibited the highest 4-PFS and FFS after 24-h-immersion among three hybrid composites, whereas after 1-year-immersion, all three composites showed almost identical 4-PFS and FFS statistically. A strong correlation was observed between 4-PFS and FFS of the machinable composite and three hybrid resin composites.

Keywords: flexural strength; fatigue strength; machinable composite; hybrid composite; esthetics

INTRODUCTION

Dental porcelain have been used successfully for esthetic crowns and fixed partial dentures. The unique properties of porcelain, including biocompatibility, stability, and optical qualities have resulted in a wide variety of accepted applications. However, porcelain restorations require much more laboratory work for fabrication and can lead to excessive wear of opposing teeth or restorations. Recently, prefabricated composite material for CAD/CAM and hybrid resin composites containing ceramic particles were developed for esthetic restorations.¹ Such materials were further improved for application in stress bearing areas. Therefore, the mechanical properties, and above all fatigue resistance are important factors for restorations supporting masticatory loads.

It is recognized that many failures of materials used in the mouth may be fatigue-related.² The loss of substances by fracture or wear may be due to the growth of cracks at relatively low levels of stress. The life expectancy of dental resin composite and ceramic restorations and luting cements depends, among other factors, on occlusion and articulation, because they generate wear and induce cyclic loading. It is apparent that the response of restorative materials to cyclic stress is an important consideration in understanding and improving clinical performance. Fatigue testing is a standard engineering procedure used to determine the endurance limit of materials and structures.

During a fatigue test, a material or structure is cyclically loaded with a decreasing range of stress and the number of cycles to failure is recorded for each stress set. The stress level at which failure does not occur after a predetermined number of cycles represents the fatigue or endurance limit of the investigated material or structure.³ During mastication, restorations and luting cements are repeatedly stressed in the oral environment and must endure for a long period.⁴ The strength of materials is usually tested in a static strength test, in which a load applied is increased in a single cycle to failure. However, this method does not reflect durability, and the analysis of dynamic tests has therefore become a subject of research.⁵ This is especially important for materials used in esthetic restorations where exposure to repeated heavy forces is likely.

Only a few studies have been performed regarding the flexural fatigue behavior of dental restorative composites⁶⁻⁸. However, no comparative data of flexural and fatigue strength on the most recent materials such as machinable and prosthodontic composite materials for esthetic restorations are available in the literature. The purpose of this study was to determine the cyclic flexural fatigue strength of a machinable and three hybrid resin composites after long-term immersion in water and to compare these values to their static flexural strengths.

MATERIALS AND METHODS

Machinable and light-activated hybrid composites

Machinable composite (GNCOM) specimens ($2 \times 2 \times 25$ mm) were prepared from CAD/CAM blocks (GN-I) using a low-speed cutting saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA). Three light-activated hybrid resin composites {Artglass (AG), Estenia (ET), and Gradia (GD)} were also prepared. Descriptions of these materials are summarized in Table I.

Preparing test specimens and the bending test

The paste of one of three resin composites was inserted into a hollow glass tube with rectangular cross section (approximately $2 \times 2 \times 25$ mm) and then cured in a xenon strobe laboratory-photo-curing unit (Dentacolor XS, Kulzer and Co., GmbH, Friedrichsdorf, Germany) on each upper and lower side for each 90 s. The specimens were briefly soaked in water and then ejected from the glass tubes, allowed to stand for 30 min at room temperature, and then stored in water at 37°C for 24 h, 6 months, and 1 year. The flexural strength (4-PFS) was then measured with a universal testing machine (DCS-500, Shimadzu Corp., Kyoto, Japan) at a cross-head speed of 1.0 mm/min. Flexural strength testing was performed in a 4-point bending mode with a span length of 20 mm and distance between loading points of 10 mm. Nine specimens of each material were

prepared for the test.

Flexural fatigue test

Twenty specimens of each material were prepared as described above and tested for flexural fatigue strength (FFS) in a fatigue tester (Higuchi Corp., Nagasaki, Japan) using a thermostatic water bath at approximately 37°C. The machine imposes a cyclic loading routine through the gradual application and removal of steel weights by the action of an eccentric cam driven by an electric motor. Three hybrid composites were cyclically loaded for a maximum of 5,000 cycles at a mean frequency of 120 cycles per min (2.0 Hz) while the machinable composite underwent a stair-step protocol with the repeats of fatigue cycles; 1, 10, 100, 500, 1,000, 2,500, 5,000, and 10,000. The flexural fatigue limits were determined by the staircase method, as described in previous articles.^{9,10}

The staircase method was employed using a 5 MPa stress increment and 20 specimens for each test. The test started for the first specimen in each group at approximately 70% of the mean 4-point bending strength. When a specimen was not fractured within 5,000 cycles, another specimen was tested at a stress level one increment higher. If the specimen fractured before 5,000 cycles, another specimen was tested at a stress level one increment lower. Each specimen was therefore tested sequentially, and the flexural load applied depended on whether the previous specimen fractured. The fatigue flexural strength in this study corresponded to the stress at which 50% of the specimens fractured within 5,000 cycles.

The means and standard deviations for the FFS were computed, and compared using two-way analysis of variance (ANOVA) and Tukey's HSD multiple comparison tests at a significance level of 0.05. The Pearson's correlation coefficient and corresponding level of significance were calculated to analyze a possible correlation between 4-PFS and FFS.

RESULTS

Figure 1 illustrates a typical staircase result of fatigue testing after 5,000 cycles for the machinable composite material (GN-I composite). The method for analyzing staircase test procedure data on the GN-I composite is shown as in Table II. The calculations of the mean and the standard deviation of FFS are given by the following equations.⁹

$$X = X_0 + d [A/N \pm 1/2]$$

$$SD = 1.62d \{[(NB - A^2)/N^2] + 0.029\}$$

X indicates the mean FFS; X_0 is the load at the lowest stress level at which the fracture or non-fracture of the specimens occurred; d is the stress increment; SD is the standard deviation. A minus sign is used in the upper equation when the FFS is obtained from fractured specimens, and a plus sign when obtained from non-fractured specimens. The lowest bending load at which fracture or non-fracture occurred was designated as $i = 0$, the next as $i = 1$, and so forth.

The number of events at the given stress level equaled n_i , A equaled the sum of in_i , and B equaled the sum of i^2n_i .

Plots between FFS of the machinable composite derived from the staircase method and the number of flexural load cycles can be generated as shown in Figure 2. A coefficient calculated from regression analysis indicated how well the data fit the curve ($R = 0.994$).

The means and standard deviations of 4-PFS and FFS for each immersion period are listed in Table III. Both 4-PFS and FFS of the GNCOM material after all immersion periods were significantly greater than those of three hybrid resin composites. The FFS results followed the same pattern as those of the 4-PFS. The ET material exhibited the highest 4-PFS and FFS after 24-h-immersion among three hybrid composites, whereas after 1-year-immersion, all three composites showed almost identical 4-PFS and FFS statistically.

A statistically significant correlation could be detected between 4-PFS and FFS of the four tested materials after each immersion period with Pearson's correlation coefficient ($R = 0.90$, $P < 0.001$) (Figure 3).

DISCUSSION

With repeated contact stress, dental restorations during mastication deform cyclically, and fatigue cracks may develop inward from the surface of the

restorations at the edge of the contact zone.¹¹⁻¹³ The loading cycles must therefore be at least partially tensile.¹³ Furthermore, brittle materials are much weaker under tension than compression. Tensile strength is therefore considered to be the more meaningful parameter for assessment of brittle materials such as composites and cements, especially in the presence of critical surface flaws.¹⁴ Thus, we selected a 4-point bending stress for the specimens in this study.

For the GNCOM material, the result indicates a clear relationship between the numbers of fatigue cycles and FFS derived from the staircase method. The FFS gradually decreased in proportion to the increase of the number of fatigue cycles, and then reached a fixed value of FFS at approximately 5,000 cycles. This investigation employed a relatively high cyclic stress for a comparatively small number of cycles. Other researchers have reported fatigue cycling of over 10,000 cycles.^{5,15,16} However, the preset of 10,000 cycles in this study is acceptable because the mean number of cycles at fracture was around 5,000 cycles or lower. Huysmans *et al.*¹⁷ also found that composites tended to fracture either early, i.e. before 10,000 cycles, or very late, i.e. after 100,000 cycles.

The frequency should lie within the limits of 0.83-2.0 Hz to give comparable chewing speeds for realistic cyclic fatigue bending tests of denture base resins.¹⁸ The use of the upper end of this range should prevent undue protraction of the test duration. A normal chewing cycle consists of a load phase (0.1-0.4 Hz)

and then a non-contact phase (0.5-2.0 Hz).¹⁹ To simulate conditions in the mouth, the composite specimens were tested in flexure using cycling fatigue corresponding to the off-on loading of a chewing cycle.¹⁵ A frequency of 2.0 Hz in this fatigue test, the upper limit of chewing frequency, was used to prevent undue protraction of the test.

This study showed a strong correlation between 4-PFS and FFS of machinable composite and three hybrid resin composites (Figure 3). It is impossible to predict how many stress cycles a restoration must withstand before failure. Theoretically, a substance will withstand an infinite number of cycles if loaded below its fatigue limit. However, fatigue life will depend on the nature of the materials, magnitude of the applied load, cyclic loading frequency, and the testing environment.¹⁰ In this study, significant reduction in 4-PFS between immersion periods of 6-months and 24-h was observed for four resin composites except for the AG material. However, this phenomenon was not seen in the FFS of the AG and GD materials. Both 4-PFS and FFS after 1-year-immersion barely decreased compared with after 6-months-immersion. For the GNCOM, ET, and GD materials in 4-PFS and the GD material in FFS, strengths after 1-year-immersion were a little bit higher than those after 6-months-immersion. Similar results of a little increase of the mechanical properties during long-term water storage were seen in other studies^{20,21}. These findings appear to be associated with a different effect of the plasticization

between brands, caused by the incoming water and the outcoming unreacted species²².

The 4-PFS of the machinable composite material tested in this study was much greater than that of other hybrid posterior composites previously reported.^{6,23} Recently, microfill composites have been shown to have a lower fatigue strength than more heavily filled materials.^{7,24} However, the microfill machinable composite evaluated in this study had a lower amount of filler than the three hybrid composites, and still had superior fatigue resistance. This would seem to suggest that the fabrication of the machinable composite material, which incorporated both heating and pressure, influenced the results. The ET material containing the heaviest filler showed the highest FFS among the three hybrid composites, whereas the three FFSs after 6-month- and 1-year-immersions were almost identical. This suggests that in addition to the contribution of filler to the matrix of the composite, filler-matrix interactions appear to greatly influence the flexural properties of the tested composites in this study. Water absorption causes filler-matrix bond failures in composites. It leaches out filler elements and induces filler failures and filler-matrix debonding, reducing the properties of resin composites.

Many factors besides mechanical fatigue can shorten the clinical life of restorative materials. Since the loads in the mouth tend to be repetitive, materials with better fatigue resistance should do better in such an environment.

Flexural fatigue behavior of resin composite materials may be useful in evaluating long-term mechanical properties.

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