Effects of Cyclic Loading on the Strength of All-Ceramic Materials

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Purpose: To investigate the effects of fatigue on the strength of materials used in all-ceramic crowns, the biaxial flexural strength of all-ceramic restorative materials was measured with precracked and laminated specimens after cyclic loading. Materials and Methods: Two types of all-ceramic systems were used to prepare specimens: a glass-infiltrated alumina core system (In-Ceram) and a leucite-reinforced feldspatic porcelain system (IPS-Empress). Monolayer and laminated disks with a diameter of about 11.75 mm and a thickness of 1.20 ± 0.05 mm were prepared. The biaxial flexural strength of the specimens that were polished and/or created with a precrack was measured. Their strength was also measured following cyclic loading. A cyclic load that was 60% of the mean breaking load of the specimens (before cyclic loading) was applied to specimens for $10^5$ cycles in 37°C water. Results: Although 20% to 30% of the polished specimen samples fractured during cyclic loading, the biaxial flexural strength of specimens that survived the cyclic loading was nearly the same as that of specimens not subjected to the cyclic loading. The strength of the alumina system decreased with the introduction of precracks, and nearly all specimens fractured during cyclic loading. The strength of the leucite system, however, did not decrease with the presence of precracks, and no fractures were observed on these specimens during cyclic loading. Conclusion: These results suggest that although the alumina system has high flexural strength, it is more sensitive to flaws and susceptible to fatigue fracture. The effect of fatigue on the leucite system appears to be low. Int J Prosthodont 1999;12:28-37.
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Table 1 Materials Used in This Study

<table>
<thead>
<tr>
<th>Classification</th>
<th>Product</th>
<th>Manufacturer</th>
<th>Type</th>
<th>Material</th>
<th>Name</th>
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<tr>
<td>Glass-infiltrated alumina core ceramic (Alumina system)</td>
<td>In-Ceram</td>
<td>Vident</td>
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<td>Vivadur Alpha Opake</td>
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<td>Ivoclar Williams</td>
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<td>Dentin-core</td>
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Table 2 Specifications of Monolayer and Laminated Specimens

<table>
<thead>
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<th>Product</th>
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<th>Specimen</th>
<th>Abbreviation</th>
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<tr>
<td>In-Ceram</td>
<td>Monolayer</td>
<td>Core</td>
<td>In-C</td>
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<td>Monolayer</td>
<td>Opaque dentin</td>
<td>In-OD</td>
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<td>In-L-C</td>
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<tr>
<td></td>
<td>Laminated</td>
<td>Tensile side: opaque dentin</td>
<td>In-L-OD</td>
</tr>
<tr>
<td>IPS-Empress</td>
<td>Monolayer</td>
<td>Dentin-core</td>
<td>Em-DC</td>
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<td>Monolayer</td>
<td>Incisal</td>
<td>Em-I</td>
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<td>Laminated</td>
<td>Tensile side: dentin-core</td>
<td>Em-L-DC</td>
</tr>
<tr>
<td></td>
<td>Laminated</td>
<td>Tensile side: incisal</td>
<td>Em-L-I</td>
</tr>
</tbody>
</table>

Cyclic Loading on Polished Specimens

Table 2 details the specifications for the specimens and testing conditions used. As the first step, a biaxial flexure test was performed without cyclic loading. It was performed according to ISO 6872. A specimen was placed on 3 steel spheres positioned 120 degrees apart on a circle (8.0 mm in diameter), and a flat-end loading cylinder with a radius of 0.8 mm was used. Polyethylene film 50 μm thick was placed between the loading cylinder and the specimen. A universal testing machine (DC5000, Shimadzu) featuring a cross-head speed of 1.0 mm per minute was used. Measurements were taken in water.

Cyclic loading was applied using the same device as in the biaxial flexure test. To avoid the application of uneven stress, a spherical indenter with a radius of 1.5 mm was used.

Loads ranging from 4.9 N to the maximum load were applied cyclically with a hydraulic testing machine (Servopulser, Shimadzu) in the form of a sine wave at 20 Hz for 10⁷ cycles in 37°C water. Before applying cyclic loading, the mean breaking load for each specimen type was determined using a biaxial flexure test, using the spherical indenter in advance. The maximum load was defined as 60% of the mean breaking load. The maximum load of each specimen type was as follows: In-Ceram monolayer core (In-C) = 261.1 N; In-Ceram monolayer opaque dentin (In-OD) = 36.0 N; IPS-Empress monolayer dentin-core (Em-DC) = 50.7 N; IPS-Empress monolayer incisal porcelain differ widely. Also, when microcracks exist in the ceramics, repeated loads propagate cracks. Therefore, it is essential that evaluations of durability investigate the fatigue behavior of all-ceramic crown materials that display microdefects and have been laminated.

The objective of this study was to clarify the effects of lamination and precrack on the fatigue behavior of materials for all-ceramic crowns using a biaxial flexural strength test following cyclic loading.

Materials and Methods

Preparation of Monolayer and Laminated Specimens

In this study, 2 types of all-ceramic systems were used to prepare specimens: a glass-infiltrated alumina core system (In-Ceram, Vident) and leucite-reinforced porcelain (IPS-Empress, Ivoclar Williams) (Table 1). By using molds designed to account for shrinkage, disk specimens with a diameter of about 11.75 mm were fired according to the firing schedule recommended by the manufacturers. Specimen surfaces were polished using a series of SiC abrasive papers (180-, 320-, and 600-grit, in that order) using Refine-Polisher (Refine Tec); the tensile side was then polished using 1-μm diamond paste. After polishing, the thickness of the disk was measured with a digital micrometer (CPM15-25, Mitsutoyo) to confirm that the disk was 1.20 ± 0.05 mm thick. In the preparation of laminated specimens, the ratio of the thickness of the core material to that of the laminate porcelain was set at 1:1 (about 0.6 mm each). Ten disks were made for each experimental condition. Two types of surfaces were prepared for comparison: in one group of specimens, surfaces were polished to minimize flaws, and in the other group, a precrack was produced on the tensile side as a fracture origin.
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(Em-I) = 38.9 N; In-Ceram laminated, tensile side, core (In-L-C) = 103.4 N; In-Ceram laminated, tensile side, opaque dentin (In-L-OD) = 67.3 N; IPS-Empress laminated, tensile side, core (Em-L-DC) = 34.8 N; and IPS-Empress laminated, tensile side, incisal (Em-L-I) = 42.4 N.

When specimens fractured during 10^5 cycles, the number of cycles that a specimen endured before fracturing was recorded. The biaxial flexural strength of the specimens that survived the cyclic loading was measured (with cyclic loading). In the laminated specimens, the measurement was performed on both sides: on the tensile side (In-L-C and Em-L-DC) for disks with a core material and on the tensile side (In-L-OD and Em-L-I) for those with laminate porcelain.

The flexural strength of monolayer specimens was determined by the following equation:

$$\sigma_m = \frac{3P(1 + \nu)}{4t^2 a} \left[1 + 2\ln(a/b) + \frac{(1 - \nu)}{(1 + \nu)} \left[1 - \frac{b^2}{2a^2} \frac{a^2}{R^2}\right]\right]$$

where: $P$ = load, $t$ = disk thickness, $a$ = radius of support circle, $b$ = radius of uniform loading at center, $R$ = radius of disk, and $\nu$ = Poisson’s ratio (assumed to be 0.25).

The flexural strength of laminated specimens was calculated by the following equation:

$$\sigma_l = \frac{6M}{t_a^2 k_{2P}} \left[\frac{E_a t_a^3}{E_b t_b^3} + \frac{E_b t_b^3}{E_a t_a^3} \left(1 - \nu \left[1 - \frac{b^2}{2a^2} \frac{a^2}{R^2}\right]\right)\right]$$

$$k_{2P} = 1 + \frac{E_b t_b^3}{E_a t_a^3} + \frac{3E_b t_b^3 (1 + \nu)^{\frac{1}{2}}}{E_a t_a^3}$$

$$M = \frac{P}{8\pi} \left[1 + 2(1 + \nu) \ln \frac{a}{b} + (1 - \nu) \left[1 - \frac{b^2}{2a^2} \frac{a^2}{R^2}\right]\right]$$

where: $P$ = load, $t$ = disk thickness ($t_b$ = top layer; $t_a$ = bottom layer), $a$ = radius of support circle, $b$ = radius of uniform loading at center, $R$ = radius of disk, $\nu$ = Poisson’s ratio, and $E$ = Young’s modulus ($E_b$ = top layer; $E_a$ = bottom layer). The elastic modulus of each specimen type was determined by the ultrasonic vibration technique using monolayer specimens in preliminary experiments (In-C = 255.2 GPa, In-OD = 67.2 GPa, Em-DC = 67.7 GPa, and Em-I = 67.2 GPa).

Nonetheless, in the leucite system, since there was no marked difference between the elastic modulus of the core material and the laminate porcelain, the biaxial flexural strength of laminated specimens was determined with the same equation that was used for the monolayer specimens.

Cyclic Loading on Precracked Specimens

Precracks were made on specimens by the indentation method using a Vickers indenter, which is used to determine fracture toughness ($K_{IC}$). For precracks to serve as the fracture origin, they should ideally be median cracks with the same crack length in all 3 directions in any specimens. If the relationship between the indentation load ($P$) and the length of precracks ($c$) can be established $P \propto c^{3/2}$, then precracks are estimated to be median cracks. Using a Vickers hardness tester, various loads were applied for 15 seconds to each specimen with a Vickers indenter from 3 to 10 N with a 1 N interval. Indentation was applied at 2 locations on each specimen, and the vertical and horizontal lengths of each crack were measured ($c$ value). Measurements were taken immediately after indentation. The relationship between the indentation load and the crack length ($c^{3/2}$) was determined. The linear regression for the core material for the alumina system was $c^{3/2} = 26.67P - 15.87$ ($r^2 = 0.99$), and that for the core material for the leucite system was $c^{3/2} = 39.30P - 1.13$ ($r^2 = 0.97$), which establishes the relationship of $P \propto c^{3/2}$ and suggests that these cracks were median cracks. As a result, a load equivalent of 25 µm, the smallest common value of $c$ for the alumina and leucite systems, was applied to introduce precracks (5.3 N for the alumina system and 3.2 N for the leucite system). By using the same method as for the polished specimens, the specimens were then subjected to a biaxial flexure test and cyclic fatigue experiments. The maximum loads of cyclic loading applied to each specimen were 119.8 N for In-C, 60.0 N for In-L-C, 44.0 N for Em-DC, and 33.3 N for Em-L-DC.

Furthermore, in an attempt to simulate clinical conditions, the same degree of maximum load was applied to the 2 systems. The maximum load (33.3 N) of the laminated specimens for the leucite system was applied cyclically to the laminated specimens for the alumina system (33.3 N equaled 31% of the mean breaking load for the alumina system).

Statistical Analyses

The results were evaluated using an analysis of variance (ANOVA) and Student's $t$ test.
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Results

Effects of Cyclic Loading on Polished Specimens

Except for laminated leucite system specimens with core material on the tensile side (Em-L-DC), fatigue fractures appeared before the completion of $10^5$ cyclic loading (Fig 1). For the monolayer specimens, 1 of 10 monolayer laminate porcelain specimens for the alumina system (In-OD) and 1 of 10 for the leucite system (Em-I) developed fractures. Three of ten monolayer core material specimens for the alumina system (In-C) and three of ten for the leucite system (Em-DC) developed fatigue fractures at points between $10^3$ and $10^5$ loading cycles. For laminated specimens, fatigue fractures appeared in 4 of 10 alumina specimens with core material on the tensile side (In-L-C), 2 of 10 specimens with laminate porcelain on the tensile side (In-L-OD), and 1 of 10 leucite specimens with laminate porcelain on the tensile side (Em-L-I). Fatigue fractures occurred at points between $10^3$ and $10^5$ loading cycles in these specimens.

The biaxial flexural strength of monolayer and laminated specimens in which fatigue fractures did not occur following the $10^5$ cyclic loading is shown in Fig 2. The biaxial flexural strength was not changed by cyclic loading.

Effects of Cyclic Loading on Precracked Specimens

In the alumina system, despite applied loads of less than half that applied to polished specimens, fatigue fractures appeared in 9 of 10 monolayer core material specimens (In-C) and in all of the laminated specimens with core material on the tensile side (In-L-C) at a point between $10^3$ and $10^5$ loading cycles (Fig. 3). These findings were markedly different from those for polished alumina specimens.

In the leucite system, fatigue fractures were seen in 2 of 10 monolayer core material specimens (Em-DC) at points between $10^3$ and $10^5$ loading cycles, but not in the laminated specimens with core material on the tensile side (Em-L-DC). These findings are similar to results for the polished specimens.

When the same load that was applied to the laminated leucite specimens with core material on the tensile side (Em-L-DC) was applied to laminated alumina specimens with core material on the tensile side (equivalent to $31\%$ of the mean breaking load of the In-L-C), no fatigue fractures were detected.

The introduction of a precrack significantly reduced the biaxial flexural strength of the alumina specimens ($P < 0.01$). The biaxial flexural strength of the precracked core specimens for the alumina
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Fig 2 Influence of cyclic loading on the strength of monolayer and laminated specimens. Cyclic loading did not affect biaxial flexural strength in specimens that survived.

Fig 3 The number of cycles for precracked monolayer and laminated specimens that fractured during cyclic loading.
System (In-C) was 215.7 MPa, and that of the precracked laminated specimens with core material on the tensile side (In-L-C) was 178.0 MPa (Fig 4).

In the leucite system, the introduction of a precrack did not affect the strength of the monolayer (Em-DC) or laminated specimens (Em-L-DC) ($P > 0.05$). As with the polished specimens, the cyclic loading did not markedly affect biaxial flexural strength.

**Discussion**

Biaxial flexure tests are more reliable than uniaxial tests, such as 3-point or 4-point bending flexure tests, in analyzing brittle materials, since maximum degrees of stress occur within the central loading area and spurious edge failures are eliminated. Accordingly, the present study applies this test and assesses the effect of cyclic loading on the strength of all-ceramic materials. If cyclic loading were applied using an indenter with a flattened tip as prescribed by ISO 6872, the edge of the indenter would come into contact with the specimen, inducing stress concentration. For this reason, an indenter with a spherical tip was used to apply the cyclic loading.

Fatigue is described as a phenomenon in which the characteristics of materials change over time under constant conditions. In brittle materials such as ceramics, White and coworkers reported that cyclic mechanical loading leads to the propagation of small cracks, causing ceramics to break at relatively low degrees of stress, and that cyclic loading is effective in predicting the strength of ceramics in clinical use. Depending on the design of cyclic loading, however, it is often difficult to determine whether fractures are caused by fatigue or by the act of cyclic loading. In this study, which was based on a report by Yoshinari and Derand, the effect of fatigue on all-ceramic materials was investigated by...
cyclically applying a force equivalent to 60% of the mean breaking load without cyclic loading. The number of cycles for fractured specimens was recorded. The results show that fractures occurred at points between $10^3$ and $10^5$ loading cycles for polished specimens and between $10^4$ and $10^5$ cycles for precracked specimens. Based on these results, the experimental conditions of the present study were considered to be appropriate for assessing the fatigue-related behavior of both all-ceramic systems.

Fractures occurred in some of the monolayer and laminated specimens during $10^5$ cyclic loading. As shown in Figs 1 and 3, cyclic loading was applied for at least 250 cycles before fracturing occurred, making it likely that the fractures were caused by cyclic fatigue. In terms of both the biaxial flexural strength and fracture surface, however, there was no significant difference between specimens without cyclic loading and those that survived the cyclic loading. These findings suggest that small flaws, which would not affect the results of conventional bending tests, greatly affect the degree of fatigue placed on ceramics by cyclic loading.

Fatigue fracture was observed in only 3 of 10 polished disks but in 9 of 10 precracked disks for the alumina system, thus suggesting that precracked alumina specimens are greatly affected by fatigue. On the other hand, precracks had no marked effect on the strength of the core material for the leucite system. Cracks initiated at the precrack in 9 of 10 alumina disks and only 3 out of 10 leucite disks, so the sensitivity to precracks differed between these 2 systems. In the alumina system, strength is increased by infiltrating lanthanum silicate glass into semifired alumina to reduce flaws, thus reducing the fracture origin. But the present study demonstrates that once cracks occur, the alumina system cannot prevent their propagation. Areas where precracks were introduced were etched by a mixed solution of 2.5% nitric acid and 2.5% hydrofluoric acid for 3 minutes...
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The results are shown in Figs 5 to 7. In the alumina system, cracks propagated through the grain boundary between lanthanum silicate glass and alumina particles (Fig 5). Because of the presence of cracks in bare specimens in leucite particles and matrix (Fig 6), however, the introduction of a precrack equivalent to 25 μm did not affect flexural strength. Furthermore, precracks did not propagate through the glass matrix surrounding leucite crystals (Fig 7). This can be explained in terms of the effect of compressive stress in the glass matrix caused by the differences in the thermal expansion coefficient between leucite crystals and a glass matrix, which suggests that cracks cease propagating once they reach leucite crystals. It is generally accepted that materials in which fractures occur at the grain boundary are more easily fatigued, and materials in which fractures are visible through the grain are more resistant to fatigue. To clarify differences in the mode of fracture, it will be necessary to study the Stress-Number curve of precracked specimens in detail. Nevertheless, the results of the present study shed some light on this issue.

When cyclic loading was applied to the laminated alumina specimens with laminate porcelain on the tensile side, detachment occurred in the fracture surface following a biaxial flexure test (Fig 8), suggesting a concentration of cyclic stress on the boundary between the core material and the laminate porcelain. In techniques that make use of a high-strength core such as the alumina system, the elastic moduli of a core material and a laminate porcelain differ widely, so that when pressure is applied perpendicularly to a laminated surface with the laminate porcelain (low elastic modulus) on the tensile side, cyclic stress is concentrated near the boundary, causing fractures to propagate through it. Yoshinari and Derand measured the fracture strength of crowns (in vitro) made by the alumina
system after cyclically applying 60% of the breaking load at static condition. They reported that 2 types of fractures appeared: complete fractures, which included the core, and fractures in which the core remained intact. They found that the strength of these crowns decreased greatly when fractures initiated from the boundary. Based on observations of clinically fractured crowns, Kelly et al20 and Thompson et al21 studied the cause of fractures and found that fractures originated from the internal surface of crowns made from a single layer of Dicor (Dentsply) using castable glass ceramics. It has also been reported, however, that when crowns were made with Cerestore, in which feldspathic porcelain is fired onto a high-strength core material made of magnesium and alumina spinel (about 100 MPa by a 4-point bending test31), fractures often occurred on the occlusal surface and at the boundary.21 As shown by the incidence of detachment in the present study, these reports suggest that the concentration of repeated stress on the boundary between a core material and a laminate porcelain greatly affects the durability of all-ceramic crowns. On the other hand, since the mechanical properties of the core material and the laminate porcelain in the leucite system are quite similar, the effect of stress concentration on the boundary between the core material and the laminate porcelain is minimal. In this study, the number of fractured disks was actually reduced by lamination with the leucite system. Because of the similarities in the fracture surfaces of the monolayer and laminated specimens, the effect of lamination on the strength of the leucite system is thought to be small.

Thompson et al21 reported that the fracture initiation sites of all-ceramic restorations are affected more by the location and size of the critical flaw than by the thickness of such a restoration. This partly accounts for the stable clinical performance of the leucite system even though its flexural strength is not high, as strength is unaffected by flaws, and the elastic moduli of the core material and the laminate porcelain are similar. When the same degree of cyclic loading that was applied to the laminated leucite specimens is applied to the laminated alumina specimens, all specimens lasted 10^6 loading cycles without developing fatigue fractures. The strength of crowns made by the alumina system would thus appear to be sufficient for clinical use. It has been determined, however, that the alumina system is more sensitive to flaws. Depending on the thickness of a core material and a laminate porcelain, the location of stress concentration alters, suggesting that the effect of cracks may vary greatly depending on the design and handling of crowns. The clinical use of all-ceramic materials is steadily increasing, to the point that they are even used in high-stress prostheses (eg, bridges, abutments, or posts)22-23. As shown by the introduction of precracks in the present study, it is possible that procedures such as fittings or frame adjustments may create the flaws that serve as fracture origins. Therefore, factors such as the relationship between the thickness of a core material and a laminate porcelain and the direction of stress must be carefully studied before all-ceramic crowns can be applied to clinical uses.

Since the 2 all-ceramic systems discussed herein have been clinically assessed for only a few years24-29, continued observation and study is necessary to fully determine their long-term durability.

**Conclusions**

To investigate the effects of fatigue on the strength of materials for all-ceramic crowns, monolayer and laminated specimens were made in an alumina system using a glass-infiltrated alumina core and in a leucite system using a leucite-reinforced feldspathic porcelain. Two types of specimens—polished and precracked to simulate a fracture origin—were investigated. The results were as follows:

1. Twenty percent to thirty percent of the polished specimens (both monolayer and laminated ones) fractured at points between 10^5 and 10^6 loading cycles, indicating that the fractures were caused by fatigue. The flexural strength of specimens that survived the cyclic loading was nearly the same as that of specimens not subjected to cyclic loading.

2. The flexural strength of the precracked alumina monolayer specimens was 215.7 MPa, and that of the laminated specimens was 178.0 MPa. While these values were significantly lower than those of the polished alumina specimens, the flexural strength of leucite specimens was not markedly affected by the presence of precracks.

3. Cyclic loading on precracked monolayer specimens led to fatigue fractures in most specimens made in the alumina system, but specimens made in the leucite system were hardly affected in terms of flexural strength or number of fractured disks. Although fatigue fractures developed in all precracked laminated alumina disks, none appeared in the precracked laminated leucite specimens.
Acknowledgments

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References


Literature Abstract

Whiplash injuries of the temporomandibular joint in motor vehicle accidents: Speculations and facts.

It has frequently been postulated in the literature that so-called whiplash injuries may lead to various types of lesions of the temporomandibular joint (TMJ). This review article with 95 references maintains that this is a questionable statement. After a discussion of the pathogenesis, pathophysics, and pathology of some TMJ diseases and analyses of the biophysics of the mandibular locomotor system, it is concluded that TMJ whiplash injury does not exist as a single and independent disease entity caused by motor vehicle accidents. If TMJ disc displacement and inflammation are present after a whiplash injury, it most probably existed before the accident, and subjective complaint of TMJ/facial pain may be a result of a preexisting TMJ disorder or a cervical condition associated with the accident. The authors assert that the proponents of a TMJ/few whiplash injury have never provided any evidence supporting their contention. For the suggested injury to the TMJ to occur, the biologic and physical laws would have to be suspended.


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