Fatigue Protocol Development and Testing of Cemented, Bilayered, Ceramics

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Fatigue Protocol Development and Testing of Cemented, Bilayered, Ceramics

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1. BACKGROUND

Introduction

Modern society is pushing standards of beauty for everyone. This cultural pressure translates into an increasing demand for highly esthetic dental restorations. All-ceramic crowns are definitely able to satisfy this demand. However, many dentists are still rightly concerned about the longevity of such restorations, since catastrophic clinical failures cannot yet be predictably avoided. In vitro test methods involving load application via small radius balls \((r < 10 \text{ mm})\) have been utilized in attempts to understand the mechanism of failure of all-ceramics crowns and consequently to improve their characteristics (Scherrer et al., 1996; Strub and Beschnidt, 1998; Castellani et al., 1994; Scherrer and de Rijk, 1992; Philps and Brukl, 1984; Ferro et al., 1994). Unfortunately, the vast majority of such tests create failure conditions that differ markedly from those reported in clinical failure (Kelly, 1999). Even the more sophisticated fracture mechanics approaches still appear to ignore clinical failure “boundary conditions” established from the findings of fractographic studies by focusing their attention on occlusal contact damage as a failure origin (Lawn et al., 2001; Jung et al., 2000; Lawn et al., 2002).

One key concept in the development of a more realistic testing, is the reproduction of the damage responsible for the clinical failure of the all-ceramic crowns. From several clinical reports, it appears that the catastrophic failure manifests itself as a crack involving the core of the crown with the consequential exposure of the abutment.
tooth, i.e. a full-thickness crack (Segal, 2201; Scotti et al, 1995, Haselton et al, 2000).

Chipping of the veneering surface has also been reported; however, this damage could be easily adjusted by simply smoothing and polishing the localized area of fracture and the restoration most of the time does not need to be replaced. Figures 1-3 show different damage that can involve ceramics; Fig. 1 illustrates the typical damage obtained during traditional laboratory tests: the occlusal surface of the specimen seems completely crushed, and the creation of multiple fragments (almost like powder) is evident. The latter figures (2,3), instead, are examples of clinical failure; the catastrophic fracture leads to the exposure of the abutment with few pieces (generally two) being created.

Figure 1. Fractured molar shaped specimen. Many fragments are visible and the loading surface appears crushed where it was loaded with a small ball indenter.
Figure 2. Clinical failure of a Dicor® crown. Courtesy of Dr. Susanne Scherrer.

Figure 3. Clinical failure of a Cerestore® (Johnson & Johnson) molar crown. The abutment under the fractured restoration is visible. Courtesy of Dr. Stephen Campbell.
Figure 2 shows the damage of an all-ceramic crown fabricated from a single layer ceramic (Dicor®, Dentsply, Int.). The starting point for the present study was previous work of Hunter and Kelly, 2000 where new guidelines were established for the creation of a more realistic in vitro test for a single layer dental ceramic system. This present study extended this work to a bilayer, core and veneer, ceramic system (In-Ceram, Vita Zahnfabrik, Germany) and evaluated if the results could be compared. Such behavior is expected, since clinical failures of both single-layer and bi-layer crowns appears to be similar (Kelly et al., 1989, 1990; Thompson et al., 1994). Figure 3 is an example of clinical failure from such a bi-layer crown system.
Brief Review of Previous Study

Since this project extends the test protocol developed from a previous work by Hunter and Kelly (2000), and that work remains published only as an abstract, a brief review of that experiment is included here. This description is taken (with permission of the authors) from an unpublished manuscript:

“Eighty-three porcelain crowns (OPC, Jeneric/Pentron) were bonded to specially designed molar replicas machined from dentin analog material (glass cloth filled epoxy), stored dry or in water (37°C, 10 weeks) and loaded beneath a 3 mm diameter stainless steel piston (having a radius of curvature of app. 0.5 m). Both static and cyclic tests were performed. Static failures were detected using acoustic emission and cyclic failure loads (1 million cycles) were determined using a staircase sensitivity test starting at 100 N with a step size of 100 N. Following completion of cyclic loading, the crowns were examined for subsurface crack formation by transillumination. If cracked, the next specimen was cycled at a lower load. If the crown did not fail, the subsequent specimen was cycled at a higher load. Loads during this testing ranged from 400 N to 625 N. All crowns and discs, tested either statically or cyclically, were examined for contact surface damage (e.g., Hertzian ring cracks) on a metallurgical microscope at 10x under Nomarski interference contrast (Nikon Epiphot, Nikon Instruments Div., Garden City, NY). No contact damage was noted.”

One of the purposes of this present study was to reproduce in vitro the same type of damage that has been identified from retrieved fractured clinical crown (subsurface
cracks), keeping the range of fracture loading in the physiological limits of the oral cavity, and introducing some fundamental variables such as broad occlusal cyclic loading, wet environment, and appropriate dentin analog material. The analysis of the Hunter and Kelly (2000) results showed that the presence of water was related to lower failure loads: "Wet storage alone, in fact, reduced failure loads significantly (1270 ± 181 N) compared with dry static or dry cyclic values (app. 1600 N) (ANOVA, p = 0.003; 95% Duncan); wet storage plus wet cyclic loading reduced failure loads to within clinical function (350 ± 273 N). This work demonstrated that a clinically-relevant failure crack system could be produced in leucite-reinforced ceramic crowns under cyclic loading."
Test Developmental Rational

Any properly designed in-vitro test should purposely develop (and analyze) the flaws and the stresses responsible for clinical failure behavior. Ideally, every variable potentially responsible for failure-initiating flaws and clinical stress states should be accurately simulated in the laboratory setting. Such variables include: ceramic(s) processing methods, crown geometry, laboratory finishing and clinical adjustments, silane treatment, etching, bonding, luting medium, water sensitivity, and loading conditions; all of which are likely to play important roles in the longevity of dental ceramics restorations.

Even though, it is difficult to control for all potential variables at once, intelligently designed laboratory protocols should always try to capture the most influential variables as part of simplified experimental procedures. For example, the surface area and volume of the specimen should be similar to that of a real crown to recreate the type and distribution of processing flaws and stress states produced during testing (e.g., bend bars may deviate significantly on both counts). The support used should share physical characteristics with the dentin (e.g., similar elastic modulus), since interfacial stresses appear to be important in clinical failure. In vitro specimens should be cemented, since clinical survival data strongly suggests a “cement effect” (Malament and Socransky, 1999). Applied loads should be in the range that teeth encounter in normal intra-oral function(s). Finally, a wet environment should be created to reproduce the
presence of the saliva and the possible presence of water seeping through the dentinal tubules in case of vital teeth.

Traditional failure tests of all-ceramic crowns generally involve loading the occlusal surface of fixed units with a small ball or the curved incisal edge of anterior prostheses against a flat compression platen (example in fig. 4). This would seem to be a reasonable way of simulating functional contacts. However, both of these experimental procedures can generate extremely high contact stresses.

![Image](image_url)

**Figure 4.** Specimen shaped as an incisor crown loaded under a small radius steel indenter (Dickinson et al., 1989).

A review of the literature on mastication shows, instead, that teeth are subjected to very low contact loads during function (60 and 250 N). Higher forces are only reached and maintained briefly (500-800 N) during parafunctional behaviors such as clenching or grinding (Bates and al., 1976). Higher biting loads can be also obtained on molar teeth.
then on other teeth in the arch; however, they never reach the range of values developed in the traditional tests. Resulting contact stresses are also determined by the surface area in contact (i.e. at wear facets) and the number of teeth and facets in contact. Actual intra-oral contact stresses have been measured, and were reported to be approximately 40 MPa (Hidaka et al., 1999). Most interestingly, contact stresses do not rise much between half maximum and maximum bite force, presumably due to a protective mechanism involving the tipping of teeth and recruitment of new contacts (Hidaka et al., 1999).

The comparison between the previous data and the failure loads reported by traditional testing (often 1500 N – 5000 N) shows clearly that the latter loads are unrealistically too high. One prerequisite for proper laboratory testing is the creation of appropriate contact stresses in order to avoid non-clinical damage. Extremely high failure stresses lead to damage that is distinctly different (Kelly 1999) from the cementation surface cracks reported from retrieved clinical crowns (Kelly et al., 1989, 1990; Thompson et al., 1994).

Clinical evidence indicates that the majority of fractures that occur in ceramic prostheses do so after a period of many years. Such failures generally are not related to an episode of acute overload but result from fatigue failure (high numbers of relatively low loads). It has been calculated that in one day teeth could be subjected to 1000-1400 masticatory cycles. Consequently, to reproduce in vitro a more realistic oral environment, cyclic type of loading should be selected. Traditional tests, on the contrary, load the samples statically (one load cycle) until failure occurs. This technique certainly
has its advantages, since in few seconds the test is performed. A more sophisticated apparatus and longer time to complete each experiment are needed if, instead, a cyclic loading approach is selected. Finally, every cyclic loading is defined by its frequency (along with wave form and upper/lower loads). Some authors believe that to simulate about 1-2 years of fatigue on a prosthetic restoration in the oral cavity, a frequency of 1 Hz for a total number of one million cycles should be used for each sample (Wiskott et al., 1995). Using these guidelines, approximately one week should be invested just to test one specimen, significantly increasing the time of the total experiment. The possibility of using a higher frequency and obtain similar fatigue effect has not been investigated in the dental literature.

In the attempt of achieving the closest simulation of the oral cavity, the direction of loading could play a fundamental role. As human beings do not chew in a vertical mode, uniaxially loading the tooth does not occur alone. A piston loading the same spot even in a cyclic manner, closely though not perfectly reproduces the true effect. Very expensive machines are trying to move the ceramic sample in different positions while the indenter load it at different angle each time or vice versa. However, not every laboratory can afford these high technologies. Fortunately, loading similar to that occurring at tooth wear facets can be simulated in vitro, even though in an uniaxial direction, and still the results could be considered acceptable, as long as the total load is in the range of the physiological values. In further defense of this approach, every off-axis load will have an axial component (from which failures principally derive). To
validate this statement, the type of damage introduced by the apparatus should be similar to that reported by the examination of retrieved clinical crowns.

Some brief review of the mechanical properties of ceramics is helpful in understanding the rationale of the present project. Ceramics are brittle materials. When stress is applied, they show almost no elastic deformation before failure (less than 0.2%). Due to their inability to reduce tensile stresses at the tip of the cracks, ceramics are much weaker under tension than under compression. Consequently, areas of tensile stresses should be the areas where they fail. While surface of the ceramic sample in contact with the indenter is under compression, ceramic peripheral to the indenter is under tension. These tensile fields around a blunt indenter (small ball) can lead to Hertzian cone cracks and around a sharp indenter can create radial and lateral cracking (Lawn, 1993b). Neither of these crack systems has been reported to be involved in clinical failure (Kelly et al., 1989, 1990; Thompson et al., 1994). As a result, any damage at this level should be considered as an artifact.

Another surface under tension, during loading of a supported layer of ceramic, is the internal one in contact with the support base and the cement (cementation surface). Clinically, a stress state, as created by the broad load distribution found at a wear facet, can lead to crack formation and ultimately failure due to a crack originating from the high tensile stresses at the cement interface (Anusavice and Hojjatie, 1992; Kelly 1999). This type of crack is also consistent with those described from the study of clinically failed all-ceramic restorations, as shown in Fig. 5 (Kelly et al., 1989, 1990; Thompson et al., 1994).
Figure 5. SEM image of a fractured Dicor ® crown. The arrow indicates the failure initiation from the cementation surface (Thompson et al., 1994).
Figure 6. Graphic representation of the results of a finite element analysis for a porcelain crown bonded to dentin with an 80 μm layer of resin cement and bluntly loaded on the occlusal surface (art work courtesy Dr. Kelly, University of Connecticut).

The highest tensile stress develops in the ceramic at its interface with the cement as is illustrated in Fig. 6 from finite element calculations (Ellert and Kelly, 1997). It is at this level that the initial crack starts. Traditional tests usually introduce in the sample damage at the level of the loading surface (Hertzian cone cracks) as a study of tested canine specimens demonstrated (Harvey and Kelly, 1996)(Fig7). Additionally, such contact damage can only be created using extremely high loads in such “crunch the crown” type of testing.
Therefore, traditional tests are evaluating the wrong flaw type, population and stress state; creating contact stresses and local contact damage that are not seen clinically (Harvey and Kelly, 1996; Kelly, 1999). A properly designed failure test should evaluate flaws inherent to the material or that are present as the result of the fabrication and finishing process (including clinical). Little valuable information is obtained by studying failures resulting from surface flaws introduced by the testing apparatus.
In designing the in vitro experiment, extremely important is the type of material selected for support of the ceramic during loading. Clinical failure stresses are thought to result from the mismatch in elastic properties between the ceramic, cement and dentin (Fig. 6). Therefore, the elastic modulus of the support is an important experimental variable and should be as close as possible to human dentin. In the traditional tests, often, the die’s modulus of elasticity is much higher then dentin. As reported by some authors (Harvey and Kelly, 1996; Kelly 1999), if the stiffness of the material supporting the crown decreases, the tensile stresses increase at the cementation surface (at a constant load). Since in the traditional tests the supports for the ceramic specimens are often made of metal, the samples is set to sustain higher load before fracturing due to the lower tensile stresses at its inner surface (for any given load). In the selection of the material for the support, the previous developmental work (Hunter and Kelly, 2000) should be considered, where blunt contact stress-strain curves were measured over an elastic region for an analog material (woven glass-filled epoxy) and hydrated dentin. Blunt contact stress-strain slopes for both materials were linear, indicating purely elastic behavior ($R^2 = \text{analog material } 98\%, \text{dentin } 91\%; p < 0.001)$. Elastic moduli for analog material ($4.2 \pm 0.25 \text{ GPa})$ and dentin ($3.3 \pm 0.50 \text{ GPa})$ were indistinguishable (t-test for regression lines; $p > 0.1$). This woven glass-filled epoxy appears to be a reasonable choice as a dentin analog to support the disks.

Dentin analog materials used in laboratory testing should also be able to form equivalent bonds to resin cement, as does dentin. To examine whether a cement bond
could be formed to the analog material identified above, in the previous developmental work hydrofluoric acid was used on the analog surface to attack the woven glass filler with hopes of creating micro-mechanical retention and a silane-coupling agent was used to prepare any exposed glass for bonding. Under these conditions, the bond to either wet or dry analog materials appears to be in a range to allow the simulation of dentin bonding, as seen in the Table 1

Table 1. Resin cement bond strengths to analog material and dentin (line indicates that do not differ significantly, ANOVA, 95% Duncan) (Hunter and Kelly, 1999).

<table>
<thead>
<tr>
<th>Condition</th>
<th>Shear Bond Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(mean and one standard deviation)</td>
</tr>
<tr>
<td>Dentin (wet)</td>
<td>6.5 ± 1.6</td>
</tr>
<tr>
<td>Analog (wet)</td>
<td>9.85 ± 0.5</td>
</tr>
<tr>
<td>Analog (dry)</td>
<td>9.1 ± 2.8</td>
</tr>
</tbody>
</table>

Even though some of the all ceramic restorations (e.g. Procera, In-Ceram) cannot be bonded, due to the inability to etch their core, the potential for bonding the support to the ceramic could be very useful to further investigations regarding whether bonding is an important variable in the longevity of these restorations.

The role(s) that water may play in clinical failure remain largely uninvestigated. Water is well known to weaken dental ceramics loaded under either static or cyclic...
conditions (Sherrill and O’Brien, 1974; Jones and Sutow, 1987; Myers et al., 1994a, 1994b). Water may also significantly affect the integrity of resin cements (Drummond and Savers, 1993). Water may act by (i) plasticizing the cement, (ii) dissolving fillers or matrix phases, and (iii) hydrolyzing silane coupling agents; all of which may affect cement properties as well as bonding characteristics.

Wet storage alone reduced failure loads significantly (1270 ± 181 N) compared with dry static or dry cyclic values (app. 1600 N) (ANOVA, p = 0.003; 95% Duncan); wet storage plus wet cyclic loading reduced failure loads to within clinical function (350 ± 273 N) (Hunter and Kelly, 200). While water decreased failure loads in the static test, the important finding involves the combination of cyclic loading in the presence of water. Under this test condition, mean failure loads are well within a clinically meaningful range (Hunter and Kelly, 2000). Cyclic loading implies that stress fatigue could develop microscopic cracks that eventually weaken the restoration and lead to its failure. Failure under cyclic conditions may involve the same mechanism(s) as under static, but additional damage accumulation resembling fatigue can also be observed following high number of low loads, that not seen under a single high static load (Xu et al., 1995). Under the dry conditions used in the previous study, it does not appear that cyclic loading alone introduced any subsurface damage. Cracking was not observed in any crown following one million cycles at up to 800 N and their static failure loads were unaffected as well. It remains to be determined whether the much lower mean wet-cyclic failure loads measured in the Hunter and Kelly (2000) study can be explained simply (based on
time under stress) or whether involved additional damage mechanisms became active (fatigue damage accumulation, de-bonding, cement degradation).

Fatigue

Fracture resulting from the application of low cyclic loads is known as fatigue fracture. In many materials, cyclic loading can either enhance or reduce the time to failure compared to an equivalent static load, an influence known as the fatigue effect. It would appear that a fatigue effect was demonstrated for the bonded ceramic crowns discussed above (Hunter and Kelly, 2000). It remains to be determined whether this effect (significantly lower fracture loads under wet, cyclic loading) was solely due to an influence on crack propagation within the ceramic, to damage accumulation in the cement, or synergistic effects. Processes that lead to a fatigue effect are well known for metals, and could be a factor in the lifetimes of ceramics and brittle polymers (e.g., dental cements). In the case of ceramics the superimposition of cyclic loads can lead to differences in crack growth rates and lifetimes (Suresh, 1998). In brittle polymers, cyclic loads can lead to crazing resulting in cyclic softening. This may be relevant for future studies since many dental cements are brittle polymers. Permanent deformation ahead of propagating cracks in brittle plastics (involving crazing or microcracking) can lead to modes of crack initiation and growth not seen under monotonic loads (Suresh, 1998a).

Aluminum oxide (generally fine grained) and transformation-toughened zirconium oxide are the two principal structural ceramics used in fixed prosthodontics.
In-Ceram core material has many properties that are similar to aluminum oxide. Fatigue effects in aluminum oxide have only been acknowledged relatively recently, where fatigue lifetimes are found to be shorter than predicted solely due to environmental factors (i.e., chemically assisted crack growth in the presence of water) (Roebben et al., 1996). While the basic properties of In-Ceram alumina tested under static loads (strength and fracture toughness) are equivalent to polycrystalline alumina, its microstructure is quite different in ways that may differentiate it during cyclic loading. Although much less well studied than for alumina, fatigue crack growth has been documented under tensile cyclic loading for transformation-toughened zirconia (Swain and Zelizko, 1988; Sylvia and Suresh, 1989; Dauskardt et al., 1990), a material of definite interest for future work. For both types of ceramics, crack propagation rates can be influenced by specimen geometry and crack size.
**Metal-ceramic (PFM) versus All-ceramic Systems**

Recently, a considerable amount of interest has developed regarding the use of all-ceramic crowns. This may be attributed in part to the following reasons. As anterior restorations, ceramics have potential for superior esthetics over PFM crowns. In contrast to PFM crowns, all-ceramic crowns do not have an alloy component and hence eliminate the unwanted opacity caused by the metal substructure or visible metal margins. Furthermore, the use of all-ceramic crowns may avoid some of the confusion for many dentists regarding the selection of alloys for PFMs. While the high-gold-content alloys are relatively expensive, the alternatives may be less advantageous posing risks such as metal allergy, bond failure, or porcelain discoloration.

Due to many disadvantages related to the presence of the alloys in PFM crowns, a new group of high-strength ceramic materials with improved physical properties has been developed in recent years. One innovative method to increase the durability of porcelain is to replace the metal undercasting with a high strength ceramic core to support the feldspathic porcelain. Despite their drawback as being more opaque than porcelain, this type of bi-layer ceramic present a far more pleasing optical result than what would be obtained with cast metal.
Higher Core Strength Ceramics: In-Ceram.

In 1989, the all-ceramic restorative material In-Ceram (Vita Zahnfabrik, Bad Sackingen, Germany) was introduced on the market. The In-Ceram process uses aluminum oxide (alumina) and glass in a two-step process to create a high-strength core material that is then veneered with aesthetic feldspathic porcelain.

The following steps are used in the fabrication of the substructure for VITA In-Ceram® Alumina restoration. A dispersion of alumina particles in water, called slip, is painted on a gypsum die. Water, flowing under capillary pressure into the gypsum die, compacts the alumina particles against the die. After that, the alumina is partially sintered at 1,120 °C to form a porous substructure where necks between touching particles are evident. The porous partially sintered alumina is then infiltrated with a low-viscosity glass (1,100 °C for 4 hours) to yield a ceramic coping of high density and strength.

The infiltration glass is lanthanum aluminosilicate. Lanthanum decreases the viscosity of the glass to assist infiltration and increases its index of refraction to improve the translucency. The rest of the crown is then formed by firing a body of porcelain over the cores by traditional firing using feldspathic porcelain. The combination of two procedures, the sintering of the alumina slip and the glass infiltration gives the material its outstanding properties. The densely packed alumina particles limit crack propagation and the infiltration with glass eliminated all porosities.
Figure 8. Sintered alumina particles (Pröbster and Diehl, 1992).

Figure 9. Glass infiltrated alumina core (Pröbster and Diehl, 1992).
In-Ceram crowns appear to be viable approaches to providing practitioners with practical, durable, and universal all-ceramic crown systems for both anterior and posterior teeth. This is particular important in light of the observations of McLean of all-aluminous porcelain crowns having only an 84.4% and 93.6% success rate (15.6% and 6.4% failure rate) for molars and premolars, respectively. The same study reported a success rate of 98.7% for canines and 97.9% for incisors (1.3% and 2.1% failure rate, respectively). No failure was recorded for 21 anterior and 40 posterior In-Ceram crowns over service lives of 4 to 35 months (Pröbster, 1993). In another clinical study, 63 In-Ceram crowns were evaluated for a minimum of 24 to a maximum of 44 months. The success rate was 98.4%, since one crown fractured exposing the abutment. The average thickness of the core was 0.48 mm (Scotti et al., 1995).
Statistical and Power Considerations.

One major goal of this project was to develop a test able to distinguish among mean cyclic failure loads in the event that meaningful differences exist based upon clinical variables; for example cement type, core ceramic type, or core/veneer thickness ratios. Additionally, if narrow ranges of data can be achieved, this test approach may prove valuable for more fundamental materials research. For developmental purposes, the capability to distinguish means 50 N apart was considered meaningful. Power to make such a distinction depends on the expected standard deviation of the means and the number of specimens tested. For the 50 N target to be practical, the standard deviation of the mean must be reduced considerably from that reported above for the predecessor project (Kelly and Hunter, 2000).

Two main factors control the standard deviation during such testing: (i) the naturally wide variability of ceramic parts during fatigue testing; and (ii) the step size chosen for the staircase sensitivity design. Providing a rich source of flaws in the surface of ceramic discs is one method of narrowing, the distribution of failure loads – i.e., using well-abraded specimens. Such an approach is fundamentally supported by three considerations. First, uniform surface damage (versus polishing) is often specified in standardized strength tests. Second, testing the ceramic in such a condition mimics manufacturer recommended dental laboratory processing and will provide realistic surfaces. Third, differences among clinical variables of interest are more likely to be meaningful than would be the case if the disks were highly polished.
The following equation represents the form of those that was used to calculate standard deviations (Natrella, 1963):

\[ SD = 1.62d \left[ \frac{(N - R)B - A^2}{(N - R)^2} + 0.029 \right] \]

where \( d \) is the chosen step size and the ratio term (containing \( N \), \( R \), \( A \) and \( B \)) is generally between 0.3 and 1.2. Thus, the standard deviation is dominated by the step size, \( d \). In order to have a reasonable standard deviation of say 50 N (see below), the step size probably needs to be around 25 N. As will be developed shortly, the initial plan was to use a limited number of disks (perhaps 5) to evaluate for the general location of the mean using a step size of 50 N. Then the definitive experiment was run with a step size of 25 N, starting at the “guess value” obtained by the 50 N preliminary evaluations.

Power analysis suggested that 15 specimens per group should be sufficient for any comparative experiment, assuming: (i) a standard deviation of 50 N (equal for both populations); (ii) equal sample sizes in the two groups; (iii) a magnitude between the means of 50 N; (iv) a type I error of 5% \((p = 0.05)\); and, (v) being able to detect a true difference with an 80% probability (type II error). Thus 15 disks per “experimental group” were initially utilized for this portion of the project (i.e., five for the initial 50 N step testing and twenty for the 25 N step size testing)
2. HYPOTHESES

1. Cementation-surface origin cracks will be introduced into the core ceramic, by cyclic loading, of veneered In-Ceram alumina disks cemented to dentin analog bases with zinc phosphate cement, and these cracks can be documented by visual inspection.

2. Coefficients of variation of approximately 10% (standard deviations of ± 50 N) can be achieved in cyclic fatigue testing of veneered In-Ceram alumina disks cemented to dentin analog bases with zinc phosphate cement.

3. Cyclic fatigue failure loads will be indistinguishable when tested at 2 Hz, 10 Hz or 20 Hz (500,000 cycles) for veneered In-Ceram alumina disks cemented to dentin analog bases with zinc phosphate cement.
3. STATISTICAL DESIGN

Portions of this project were descriptive in nature. Where comparisons were to be made, mean failure loads and standard deviations were evaluated using staircase sensitivity testing (Collins, 1981; Dixon and Mood, 1948; Natrella, 1963). The t-test was applied for comparing between independent groups using mean and standard deviation statistics calculated from the staircase sensitivity results (Schelfler, 1979). Linear regression analysis was applied to the mean failure loads as a function of testing frequency. ANOVA and a 95% multiple range test were applied for comparison among groups based on water storage times.
4. MATERIALS AND METHODS

Preparation of the Bilayered Ceramic Disks

Sixty glass-infiltrated alumina core ceramic disks were utilized for this study. The disks were made of In-Ceram alumina, veneered with VM7 window porcelain (Vita Zahnfabrik, Bad Säckingen, Germany).

![Cross-section of a 0.6 mm In-Ceram and 1.4 mm VM7 ceramic disk.](image)

**Figure 10.** Cross-section of a 0.6 mm In-Ceram and 1.4 mm VM7 ceramic disk.

Each core disk was prepared with the slip-casting technique according to the manufacturer’s recommendations (final dimensions = 12 mm diameter, 0.6 mm thick) (Fig. 10). To create the mold base for the slip-casting of the core ceramic, an aluminum disk (12 mm diameter, 0.8 mm thick) was placed on a block made of type III die stone. Polyvinylsiloxane extra light body impression material (Aquasil, Caulk/Dentsply) was then injected around each disk and a glass slab was placed on top until the aluminum
surface was visible. Once the glass slab and the aluminum disk were removed, the mold was filled with a dispersed aluminum oxide (slip). Water in the slip was drawn into the gypsum base by capillary forces, packing the aluminum oxide powder. Following ten minutes of drying, excess alumina was removed with a sharp, flat blade and the impression material was carefully removed from around the greenware disk. Alumina disks were given a first firing (initial sintering to form “necks” between touching particles) while still upon their gypsum bases (30 min. 120 °C, 2 hours 1120 °C; In-Ceramat II, Vita). After sintering, porous disks were placed on a platinum sheet and glass infiltrated to achieve final density (30 min. 200 °C, 2 hours 1100 °C; In-Ceramat II, Vita). For one initial batch (batch 1), excess infiltration glass was removed with a coarse grain diamond instrument and no glass control firing was performed (discussed below). Disks were submitted to a ceramic machinist as discussed below. For a second batch (batch 2), infiltration glass excess was removed as part of the ceramic machining (no coarse dental diamond instruments used) and disks were given a glass control firing following machining (10 min. 1000 °C, Vacumat, Vita). The final thickness of preliminary core disks was approximately 0.8 mm in consideration for the final polish thickness of 0.6mm. Final specimen thickness (0.6 mm) and surface finish (600 grit silicon carbide) were subsequently obtained by expert machining and polishing of both sides of the disks (BOMAS Machine Specialties, Somerset, MA).
The surface to be veneered was treated using airborne particle abrasion (50 µm alumina at 0.3 MPa) followed by ultrasonic cleaning in acetone for 1 minute. To guarantee the appropriate thickness of the veneer, a washer of 2.2 mm depth was used to accommodate the core disk (0.6 mm), the veneering porcelain (1.4 mm) and the material eventually lost during the subsequent polishing process (0.2 mm) (Figs. 11 and 12).

Figure 11. Plastic mold used to create the veneering layer.
The veneering ceramic was condensed and fired to 960 °C according to the manufacturer instructions. A second firing cycle was needed to compensate for porcelain shrinkage. Final specimen thickness and veneer surface finish was controlled by expert ceramic machining (BOMAS Machine Specialties, Somerset, MA).

Finally, the veneering surface was polished on a polisher wheel with alumina oxide particles of 0.03 μm (Polimet polisher, metallurgical apparatus, Buehler Ltd, IL USA), while the core surface was sandblasted (50 μm alumina oxide particles at a pressure of 3 MPa). Before the cementation, the disks were cleaned in an ultrasonic cleaner with acetone for 1 minute and dried.
Preparation of the Supporting Substrates

Bases to support ceramic disks during testing were machined from rods of epoxy-glass cloth material (NEMA grade G10, International Paper, Hampton, SC). The cementation surface of the base was sandblasted (50 μm alumina oxide particles at a pressure of 3 MPa) and air-blasted cleaned. To create a wet environment, these dentin analog structures were modified by creating small channels to allow water to access the cementation surface, simulating the condition established by dentinal tubules. Five micro-channels were obtained with a cobalt HS drill #73 starting from the central well to the cementation surface on the opposing side (Fig. 13).

Figure 13. Five micro-channels were drilled into each base using a cobalt HS drill from the central well to the cementation surface.
To facilitate the flow of water, four lateral channels emanating from the central well were also created. The micro-channels were filled with accessory gutta percha points (size 40 K) so that they remained open following cementation (Fig. 14).

**Figure 14.** Five gutta percha points in the microchannels of the base, before cementation of the disk. The tips emerging from the cementation surface will be trimmed away.
Specimen Cementation

Each disk was cemented on a dentin analog base. A type 1 zinc phosphate cement (Fleck, Mizzy Inc. Cherry Hill, New Jersey) was mixed in a constant-temperature room (20°C), and the glass slab was cooled at approximately 15°C by placing it in a basket full of ice. The powder/liquid ratio was 0.3 g / 5 drops and the mixture was spatulated for 50 seconds. A micrometer was used to control the cement layer thickness (50 μm) for all specimens (Fig. 15). Modifications to the micrometer were made to accommodate samples using a custom plastic jig. Polyvinyl siloxane was used to fasten the jig to the micrometer. Before cementation, the disk and base were placed in the jig and the micrometer was calibrated to zero. Once zeroed, the disk was removed, and zinc phosphate cement applied to the core surface. The specimen was then placed over the base and the micrometer set to 50 microns.

Figure 15. Micrometer used for cementation.
After two minutes of the initial set, the jig assembly was removed from the micrometer and through a hole in the jig, gutta percha, used to obdurate the micro-channels of the base during cementation, was removed. The sample was then set aside and allowed to rest for 24 hrs to ensure complete setting of the cement (Figs 16a-b).

Figures 16a - 16b. Jig assembly and removal from it of the gutta percha points.
Water Storage

Each cemented sample was stored at room temperature in de-ionized water for at least two weeks. In order to eliminate the air bubbles from the five micro channels of the base, a #30 endo file was inserted in the micro-channels to remove trapped air, allowing water to penetrate into the channels. Specimens were then placed disk-face down in a beaker of de-ionized water covered with aluminum foil. This was done to ensure that water would remain in the central well of the base while transporting the specimen from the beaker to the testing machine (Fig 17).

Figure 17. Water storage of the cemented disks face-down in the beaker.
Fracture Loading

Each cemented specimen was accommodated inside the testing machine (MTS 858 MiniBionix II) (Fig 19). The functional unit consisted of a water chamber placed on top of the load cell and an indenter connected to the actuator (Fig 20). The water chamber accommodate the specimen and it was filled with de-ionized water. Attention was paid to replace the water that eventually evaporated during the testing. After every test, the chamber was emptied, cleaned and refilled with fresh water. Disks were centrally loaded using a 3 mm diameter piston. A sheet of polyethylene (0.1 mm thick) was placed between the piston and disk to further reduce contact stress concentrations.

In the pilot study, three different indenters made of stainless steel, aluminum, and a woven glass/epoxy resin were tested before selecting the final piston. The use of the first two was discontinued, since damage of the loading surface of several specimens was recorded (see Fig. 23). The piston chosen for the final test was made of the epoxy-glass material (NEMA grade G10, International Paper, Hampton, SC), the same material used by Hunter and Kelly (2000) and this project to fabricate the specimen support bases (Fig18).

Figure 18. Final indenter selected made of epoxy-glass material.
Figure 19. MTS 858 MiniBionix II testing machine.

Figure 20. Close view of the functional unit of the MTS machine.
Specimen Alignment

Due to the difficulty of aligning the disk precisely over the base during the cementation process, a device was constructed to realign the disk under the indenter of the MTS testing machine (Fig 21). This plunger-shaped device was made with a stainless steel rod oriented perpendicular (in a dental surveyor) and centered over a disk. A GC resin base (GC Corporation Tokyo, Japan) was created to fit the circumference of the disk. Each time a specimen was loaded in the MTS machine, this device in combination with the ability to slide the lower chamber of the MTS machine, was used to ensure proper alignment with the piston (Fig 22).

Figure 21. Alignment device placed on top of the disk and the base inside the testing chamber.
Figure 22. The indenter and the rod of the alignment device do not correspond perfectly. To center the specimen under the piston, the lower chamber of the MTS machine needs to be moved slightly to the right.
Cyclic failure loads (500,000 cycles, at 2, 10 Hz and 20 Hz) was evaluated using a modified two-stage staircase sensitivity design. In the first phase, five disks were tested, starting at 500 N with a step size of 50 N, to provide guidance for the initial load value in the second (definitive) phase. The second phase began at the load determined in the first phase, and preceded with approximately 15 disks at a step size of 25 N. All disks tested cyclically were examined for contact surface damage (e.g., Hertzian cone cracks) Data from disks demonstrating contact damage were excluded from statistical consideration.

Disks were debonded from their bases via an impact load to their periphery. All disks were examined by optical microscopy (under transilluminated lighting) at 10x magnification (Nikon SMz 800, Nikon, Japan). Fracture surfaces of selected disks were examined by scanning electron microscopy to ascertain failure origins. Scanning Electron Microscopy was conducted using a JEOL JSM-5300 field emission SEM with digital image capture in the Biomedical Materials Group Facility at the National Institute of Standards and Technology, Gaithersburg, MD.
5. RESULTS

Disk Dimensions

The thickness of five randomly chosen core disks from batch 1 was measured with a digital caliper and found to be well controlled and as specified (0.6 ± 0.03 mm) (Fig 23). Final veneered disk thickness was checked for 15 randomly chosen disks from batch 2 and found to be well controlled and as specified (1.987 ± 0.02 mm).

Figure 23. Alumina core final thickness was confirmed by a micrometer.
Suppression of Hertzian Cone Cracks

Three different indenters made of three different materials (stainless steel, aluminum and resin-based composite) were tested before selecting the final piston. The following diagram (Fig. 24) shows the results obtained for the initial investigation using the stainless steel and the aluminum pistons. Cone cracking occurred frequently with stainless steel piston. One cone crack occurred with use of the aluminum piston after only 8 specimens, and attention turned to the even less stiff epoxy-glass material. No cone cracking encountered with resin-based composite piston (dentin analog material).

Figure 24. Diagram of the results of testing at 10Hz with stainless steel and aluminum indenters.
Visualization of Subsurface Cracks

Easily identified without need for transillumination or magnification. See Fig. 25. Visualization of cone cracks was enhanced by painting surface with penetrating dye, while the disk was still cemented to the support to verify the damage to the loading surface. See Fig. 26a – 26e.

Figure 25. Veneering surface of a bonded disk still cemented to the base. After cyclic loading, a subsurface crack was identified.
Figure 26a. Disk cemented to base. The loading surface is damaged (cone crack).
Figure 26b. Initial dye penetration from the damaged occlusal surface of the previous disk in Fig. 26a.
Figure 26c. Dye penetration progresses from damaged occlusal surface of disk following the defects created by the cone crack.
Figure 26d. Increased dye penetration (suggesting core-veneer delamination).
Figure 26e. Total penetration of the dye. The origin of the damage from the loading surface is confirmed.
Origin of Subsurface Cracks

All cracked disks were separated from dentin analog bases. Cracks in the cementation surface of core material had same orientation and approximate length as crack extension visualized in veneer porcelain (Fig. 27).

*Figure 27.* The disk in fig.25 was debonded and its cementation surface evaluated. The red arrow points to the subsurface crack (already visible from the veneering surface).
Crack extension from cementation surface was confirmed by application of penetrating dye (Figs. 28a-28b).

**Figure 28a.** Debonded disk showing a subsurface crack visible from the veneering surface.
Figure 28b. The dye was applied on the cementation surface of the disk in Fig. 28a. After dye penetration, a subsurface origin of the crack was suggested.
In addition, the disks where cone cracks were identified were also debonded. The core surface was always intact and the dye applied on this surface did not penetrate, confirming that the damage was starting from the loading surface. See Figs. 29a–29b.

**Figure 29a.** Cementation surface of the disk in Fig. 26a where a cone crack was identified. No visible damage of the core side of the disk is evident.
Figure 29b. Cementation surface of disk in Fig. 26a. Notice that after dye application there are no evident defects.
Three disks were randomly chosen for fracture surface analysis (SEM). This analysis found that all fracture surface features were consistent with the origin of cracks being the core cementation surface. See Figs. 30a-30d.

**Figure 30a.** SEM of fracture surfaces of both disk halves (one up and one down). Core ceramic is lighter material and porcelain is darker material. Labels refer to fracture surface features: (a) wake hackle; (b) crack branching (or twist) hackle.
Figure 30b. SEM of lower disk in Fig. 30a. Labeled fracture surface features: (b) crack branching (or twist) hackle; (d) features due to secondary fracture, by hand, of disk to expose original fracture surface.
Figure 30c. SEM of fracture surfaces of both disk halves (one up and one down). Core ceramic is lighter material and porcelain is darker material. Labels refer to fracture surface features: (a) wake hackle; (b) crack branching (or twist) hackle.
Figure 30d. Higher SEM magnification of fracture origin in Fig. 30c.
Standard Deviations and Coefficients of Variation

Mean failure loads, standard deviations, and coefficients of variation (as a percentage) are presented in the Table 2 for the four groups treated separately for statistical analysis. Over all four groups, the mean coefficient of variation was 9.3% and the mean standard deviation was 52.8 N. Focusing only on results from batch 2 disks (possibly different from batch 1 as described below) the mean coefficient of variation was 8.6% and the mean standard deviation was 50.5 N (Figs. 31-33).

<table>
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<th>CV</th>
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<td>59.49</td>
<td>11.4</td>
</tr>
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<td>10</td>
<td>603.13</td>
<td>41.04</td>
<td>6.8</td>
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<td>569.64</td>
<td>67.3</td>
<td>11.8</td>
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<tr>
<td>2</td>
<td>20</td>
<td>602.5</td>
<td>43.29</td>
<td>7.2</td>
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<tr>
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<td>52.8</td>
<td>9.3</td>
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<tr>
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<td></td>
<td>591.8</td>
<td>50.5</td>
<td>8.6</td>
</tr>
</tbody>
</table>

Table 2. Summary of means, standard deviations and coefficients of variation achieved.
Figure 31. Diagram illustrating the results of the test at 2Hz. The run-out specimens are indicated as open circles, fractured specimens are indicated with the red line, while the yellow color indicates the specimens not included in the final statistical analysis.
Figure 32. Diagram illustrating the results of the test at 10Hz. The run-out specimens are indicated as open circles, fractured specimens are indicated with the red line, while the yellow color indicates the specimens not included in the final statistical analysis.
Figure 33. Diagram illustrating the results of the test at 20Hz. The run-out specimens are indicated as open circles, fractured specimens are indicated with the red line, while the yellow color indicates the specimens not included in the final statistical analysis.
Possible Fatigue Failure Load Difference Found Between Batch 1 and Batch 2 Core Ceramic

Mean failure loads for disks made with batch 1 core (522.5 N ± 59.49) and batch 2 core ceramic (603.1 N ± 41.0 N) were both measured at 10 Hz. These failure loads differed significantly (t-test, p < 0.001). See Fig. 34.

**Figure 34.** Mean failure loads (and one standard deviation) of ceramic core batch 1 versus batch 2 (10Hz).
Cyclic Failure Loads as a Function of Cycling Frequency (2 Hz, 10 Hz, 20 Hz)

Strengths at 2 Hz (569.6 N ± 67.3 N) and 20 Hz (602.5 N ± 43.3 N) differed significantly (t-test; p < 0.05). Linear regression suggests a slight positive relationship ($r^2 = 0.68$) with approximately 5% increase in fatigue strength at 20 Hz compared to 2Hz.

See Fig 35.

![Figure 35](image-url)

**Figure 35.** Linear regression among the three testing frequency subgroups: 2 Hz, 10 Hz, and 20 Hz (all fabricated of batch 2 core ceramic). 95% confidence intervals are for the linear regression line. 2 Hz and 20 Hz means were compared using the t-test.
Water Storage Times

Water storage times for disks tested at 10 Hz (failed disks) were longer for batch 1 specimens (39 days ± 4.5 days) than for batch 2 specimens (29.3 days ± 2.9 days). Thus, the disks associated with the higher failure loads had significantly less storage time (t-test, p < 0.05). However, within each batch there was no significant difference in storage times for failed versus run-out disks (t-test, p > 0.5). Additionally, for the other disk set where a significant load difference was found (20 Hz being higher than 2 Hz) there was no difference in water storage times among any of the four groups (2 Hz failed and run-out; 20 Hz failed and run-out)(ANOVA, p > 0.08). For all batch 2 disks combined (2 Hz, 10 Hz, and 20 Hz), there was no difference in water storage times for failed (33.6 days ± 2.2 days) versus run-out disks (28.3 days ± 2.4 days). See Figs. 36-38.

Figure 36. Water storage (days) for each specimen tested at 2 Hz.
Figure 37. Water storage (days) for each specimen tested at 10 Hz.

Figure 38. Water storage (days) for each specimen tested at 20 Hz.
6. DISCUSSION

**Disk Dimensions**

The test utilized loads (for failure) as an outcome measure, stresses were not calculated. This approach is common in structural testing where specimens have complex shapes, where they are comprised of multiple materials bonded together, or where failure involves multiple mechanisms. In these situations, the actual stress in the part where failure initiates is often difficult to calculate or approximate. Examples in prosthodontics would include the failure of three-unit fixed partial dentures and the yield load for implant abutment screws. In order for test results from this type of approach to be meaningful, the dimensions of the part(s) and layer thickness should be well controlled. Core disk thickness was controlled by the ceramic machinist (BOMAS) and was found to be within the tolerances specified. Total disk thickness after porcelain veneer application were also controlled by the ceramic machinist and found to be within specified tolerances. It was important to anticipate the loss of material during the grinding and polishing and to include it into the fabrication of each sample, so after both steps (core build up and porcelain veneering) the proper thickness was achieved.
**Suppression of Hertzian Cone Cracks**

Clinical failures have never been shown to result from contact damage to the occlusal surface. It has been reported in the literature that most so-call crown “strength” tests create damage that is very unlike anything reported from the examination of clinically-failed units (Kelly, 1999). Both sharp indent and blunt indent cracking appear to be involved in these questionable protocols, with blunt indentation cracks (Hertzian cone cracks) being the most likely to occur first.

In this study a main goal was to learn how to suppress cone cracks at the piston-ceramic contact. Initial work paralleled the protocol developed at NIST (Hunter and Kelly, 2000) in using a precision-machined piston having a radius of curvature of 0.5 m (Fig.39). This piston was machined from a hardenable stainless steel that was given a hardening heat treatment following machining. Hunter and Kelly (2002) did not report any damage of the loading surface of the ceramic specimens while using such an indenter.

![Figure 39. Close view of the stainless steel piston. The end has a radius of curvature of 0.5 m.](image)
On the contrary, as illustrated in Fig. 24, in the initial phase of this protocol, using the same type of indenter, cone cracking was occurring (two cone cracks before its use was discontinued). Possible explanations for this outcome, compared to the previous work of Hunter and Kelly, include the higher loads used (app. 500 N to 650 N versus 250 N to 350 N) and a possible slight misalignment within the test machine (allowing piston edge contact with the ceramic surface). The presence of a thin layer of polyethylene sheet did not appear to fully compensate for such conditions and sharp contacts between the disk and the edge of the piston appear to have developed with both the stainless steel and aluminum pistons (Fig 40).

![Image](image_url)

**Figure 40.** A thin sheet of polyethylene interposed between the disk and the indenter inside the test chamber.
Examining the thin piece of polyethylene film at the completion of the testing, an actual contact between the piston and ceramic was likely (although localized to small areas of perforation).

Besides the radius of curvature, the elastic modulus of the piston is also an important factor in the contact stress created (Lawn, 1993). At any given load, the contact stress (with a curved surface involved) will be linearly related to the piston elastic modulus.

Therefore, pistons were machined from increasingly lower modulus materials, specifically aluminum (E \approx 70 \text{ GPa}) and the dentin analog material (E \approx 15 \text{ GPa}) (compared to steel at \approx 210 \text{ GPa}). Both of these two indenters had 3 mm diameter flat piston end. The use of the aluminum piston was discontinued when the first cone crack was recorded. The experiment progressed switching to the dentin analog material with no cone cracks encountered for during testing of more than 70 disks.

However, the dentin analog piston presented its own limitations. Since it eventually plastically deformed (splaying at tip) (see Fig. 41), the MTS machine automatically interrupted the experiment and the sample loaded had to be discharged. Unfortunately, it was difficult to calculate the number of the loading cycles that each piston could be able to withstand before failure. Some pistons lasted longer than others and only keeping track of the number of cycles did not take into consideration the total time of the tests; each loading cycle lasts longer at 2 Hz than at 20 Hz (0.5 seconds versus 0.05 seconds). To avoid the waste of specimens, frequent change of the indenters was
performed by registering the numbers of cycles each piston was involved and arbitrarily substitutes it with a new indenter after no more then four specimens. However, due to the enormous amount of time invested for the completion of the test at 2 Hz (69 hours), risks were not taken and for each specimen a new indenter was used.

Figure 41. Close view of two dentin analog indenters. Notice the deformation of the tip of the upper, plastically deformed one.
Visualization of Subsurface Cracks

This was a critical goal to achieve (as was documenting the origin of cracks produced), if this protocol was going to be extended to the testing of full crowns (instead of disks). Cracks in the ceramics used in the previous study (Hunter and Kelly, 2000) were easily visualized by transillumination through the monolithic, translucent porcelain used. In the present study, cracking was expected to begin in the opaque core ceramic and then to propagate into the porcelain layer. In order to facilitate crack visualization, a clear veneer porcelain (VM7, Vita) was utilized.

Since the indenter was fixed and the load uniaxially directed, cracks starting from the inner surface (cementation surface) were not expected to propagate to the loaded surface due to the constant presence of compressive stresses directly below the piston. The integrity of the loading surface in the fractured disks was a valid prove that the failure occurred in the cementation surface “tensile zone” of the specimen. To further prove the subsurface origin of the crack, penetrating dye was first applied to the fractured specimens while they were still cemented to theirs bases. Lack of dye penetration was a secondary indication (along with optical microscopy) that there was not contact surface cracking. The lack of dye penetration was also evident in the same disks after debonding (Figs. 42a-b). Thus, the cracks arrested within the porcelain prior to reaching the surface. However, the integrity of the loading surface by itself does not prove that the cementation surface is the origin of the failure, since still the crack could start at the interface between the core and the veneer porcelain. The next step after debonding of the fractured
specimens was to carefully analyze the core surface and look for trace of the failure crack. Additionally, three randomly selected discs were subjected to definitive fractography by scanning electron microscopy (discussed below).

Figure 42a. Veneering surface of a fractured debonded disk. A subsurface crack appears in the center of the disk.
Figure 42b. Dye applied on the veneering surface of a fractured disk in Fig. 42a. To demonstrate the integrity of the surface, the dye should not penetrate the crack.
**Figure 42c.** Veneering surface of disk in Fig. 42a. The dye was completely wiped off from the surface, confirming the integrity of the loading surface of this fractured disk.
Origin of Subsurface Cracks

Analyses of clinically failed all-ceramic crowns consistently report that failure did not occur from the contacting (occlusal) surface, but instead originated from the cementation (intaglio) surface (Kelly et al., 1989, 1990; Thompson et al., 1994). Thus, it was of critical interest to validate that the subsurface cracks, which could be visualized in the clear porcelain, were extensions of cracks originating at the ceramic-cement interface.

All of the cracked disks were removed for examination of the cementation surface. In all cases, the direction, character (e.g. a kink or bend in the crack), and the approximate length matched for the crack trace seen on the cementation surface compared to the crack seen within the clear porcelain. In no case were matching cracks not found on the cementation surface (such a finding would have raised questions about the cracks originating at the internal core-porcelain interface). Dye applied to cracks on the core ceramic surface quickly penetrated upwards into the crack within the porcelain, indicating that these were connected. See Figs 28a-b.

For three specimens containing relatively large cracks, the disks were bent (by hand) to complete failure so that fracture surfaces could be examined by SEM. See Fig. 43a-b.
**Figure 43a.** Three fractured disks with a large crack were selected for fractographic analysis (SEM).
Figure 43b. The three disks were bent by hand to a complete failure.

Scanning electron microscopic observations (SEM) of both retrieved clinical crowns and failed laboratory specimens have been extremely useful in documenting the origin and the propagation of the primary crack responsible for failure (Kelly, 1995). In the present study, a variety of fracture surface features were documented and used to confirm the failure origin. Two key features were wake hackle and crack branching (or twist) hackle. Wake hackle develops as two sides of a crack front running around a filler particle or pore reconnect slightly out of the original crack plane. A wake mark (or tail) develops in the fracture surface tracing the path of the crack front. In the case of crack branching (or twist) hackle, surface features are left as the crack front has gathered
sufficient energy to begin to develop secondary cracks (that do not fully form) creating linear discontinuities that track the path of crack advance.

Indentation damage such as seen in Fig 44, not reported from fractographic examination of clinically-failed all-ceramic crowns, was not identified in any of the three disks analyzed.

**Figure 44.** Gold-coated fracture surface of glass-ceramic canine loaded to failure against flat compression platen. The tip of the specimen has been crushed (b) and fractographic features, like crack branching (a), prove that the cementation surface (c) was not the origin of the failure (Kelly, 1999).
Instead, clear evidences of the origin of the flaw pointed towards the cementation surface. See Figs 45a–b and 46. On addition, some specimens showed fatigue striations; i.e. markings of the crack front position at steps during its growth under the cyclic loading.

Figure 45a. SEM of fracture surface; core ceramic (lighter material) is up. Labeled fracture surface features include: (a) wake hackle and (c) fatigue striations. Note that the fatigue striations in the right side are oriented perpendicular to the wake hackle while fatigue striations in the middle of the SEM are oriented parallel to the core-veneer interface.
Figure 45b. SEM of fractured surface. Labeled fracture surface features; (a) wake hackle and (c) fatigue striations.
Figure 46. SEM of fractured disk. Labeled fracture features: (a) wake hackle, (c) fatigue striations.
Standard deviations and coefficients of variation

Overall, the standard deviation encountered was approximately ± 50 N and the coefficient of variation (standard deviation divided by mean) from the four data sets in this project was slightly under 10%. These values lie well within the range typical for the strength testing (or failure load testing) of dental ceramics. Thus, it is likely that the protocol as developed and applied in this study can be used to investigate issues of interest to the dental community. Some of these issues could include: (a) comparisons between In-Ceram and other high strength core ceramics; (b) the influence of core/veneer thickness ratios; (c) variations of total ceramic thickness (c) the use of a resin cement capable of bonding to In-Ceram; (d) the influence of various dental laboratory processing steps; (e) the influence of different substructure material.

More formally, these finding relate to the power of statistical tests likely to be applied to data from testing of variables related to the use of dental ceramics. In general terms, power relates to the ability of an experiment to detect a given difference if one really exists. More specifically, power is defined as the ability to avoid making a Type II error (\( \beta \)), i.e. concluding that a difference did not exist where in fact there really was a difference. Mathematically, power is defined as \((1 - \beta)\). For an experiment to have a 90% power to distinguish a particular difference \( \beta \) would be \((0.1)\). Three main factors control power (Glanz, 1992): (a) the risk of error you will tolerate when rejecting the hypothesis of no treatment effect; (b) the size of the difference you wish to detect relative to the amount of variability in the populations; and (c) sample size.
The measure of variability in the populations used in power analysis is the standard deviation (Glanz, 1992). Power functions are calculated somewhat differently for the various statistical tests that can be applied in making strength or failure load comparisons. Since the t-test is commonly employed, it will be used here as an example. Power functions are calculated from the ratio ($\phi$) of the test effect wished to be detected ($\delta$) divided by the standard deviation (sd) (Glanz, 1992): $\phi = \delta / sd$. Thus if one wished to detect an effect size of 100 N or 50 N (e.g. the mean load increase with a bonding resin cement) $\phi$ would be either 2 or 1.

![Graph of power functions](image)

**Figure 47.** Power function graph (Glanz, 1992).
The power function graph reproduced from Glanz (1992) in Fig. 47 above, gives the number of specimens (n) needed per group to achieve various levels of power in a t-test comparing two groups for $\phi$ ranging from 0 to 2 (at $\alpha = 0.05$). Given that the degrees of freedom is used to determine the t statistic and is equal to (n-2), and that the degree of freedom under the presently developed protocol was around 30, the power function in Fig. 47 for an n of around 15 would roughly equate. Given these assumptions, the cyclic loading protocol as developed would have a power of approximately 70% to 75% to detect a 50 N effect and over 95% to detect a difference of 100 N.
Cyclic failure loads as a function of cycling frequency (2 Hz, 10 Hz, 20 Hz)

There appears to be a real, although small (app. 5%) increase in mean failure load, when cyclic testing was performed at 20 Hz versus 2 Hz. This possible trend is examined as a linear regression in Fig. 35. While it may be questionable as to whether a linear model should be applied, this analysis does illustrate the rather weak effect of cycling rate on mean failure load.

There are three main effects that cycling frequency can have on crack growth rates: (1) none (no fatigue effect); (2) a detrimental influence (a fatigue effect); or (3) a beneficial influence (Suresh, 1998a). Where a beneficial influence is seen, cycling has inhibited the growth and development of flaws in some fashion that is not seen during static loading to failure. The traditional method of examining for a fatigue effect involves either measuring the crack growth rate directly or the time to failure. What was measured in the present experiment was the mean failure load, a derivative or secondary measure of crack growth rate. Since only a secondary measure was documented and the effect was so slight, any mechanistic discussion of the origin of the effect is not very meaningful (and well beyond the scope of this project).

The major importance of this finding relates to doing the experiment and to the planning of any future work. Accomplishing 500,000 cycles at 2 Hz required 69.44 hours (2.89 days) versus 6.9 hours at 20 Hz. In any future experiments that focus on clinical lifetimes (e.g. simulating many years of function), it is not unlikely that many millions of cycles will be called for. Thus, it is very meaningful to have documented that
rapid cycling can produce results equivalent to those produced at rates closer to actual chewing frequencies

Possible fatigue failure load difference found between batch 1 and batch 2 core ceramic

Mechanical properties of ceramics are known to be dependent on processing variables, i.e. the various steps in making and finishing a ceramic part can lead to different flaw types, flaw sizes and flaw distributions (Lange, 1983, 1984). Therefore, it is likely that some processing step change explains the possible difference in the cyclic failure loads for disks made from two batches of core ceramic.

In retrospect, there are some differences in how the disks were fabricated. Batch 1 and batch 2 disks were handled slightly different during the glass infiltration process. As Fig. 48 shows, after the infiltration process, cores from batch 1 were surrounded by a conspicuous amount of excessive glass, since, before firing, each alumina core was placed on top of an excessive mix of glass. To remove such excess manufacture’s recommendations were followed using a coarse-grit diamond instrument. During this step, additional cracks could have been introduced in the core surface. However, if introduced they might also have been eliminated, since an additional thickness of 0.2 mm was included in the fabrication of the core, so after the final polish by the expert ceramic machining, the most superficial layer of both the surfaces was lost and a final thickness of 0.6 achieved.
The alumina cores in the batch 2, on the other hand, were placed first on the platinum sheet and after the glass mix was applied on top just enough to guarantee a complete infiltration with little excessive glass leftover (Fig. 49). After the glass infiltration firing, the cores of batch 2 were directly polished by expert machining without any hand grinding with rotary dental diamond instruments (same 0.2 mm thickness of loss of material during polishing was allowed-for).

When the final cores were received from the ceramic machinist, batch 1 cores were sandblasted in preparation for the veneering process. For batch 2 cores an additional firing cycle (glass control firing; 960 °C, 1 minute) was carried out, following the manufacture’s instructions. While no extra glass was “sweated out” during this extra firing (as is hypothesized by the manufacturer) the firing step could possibly have driven other beneficial processes, such as: (1) healing of grinding/polishing flaws; or (2) changing slightly the glass composition with dissolution of alumina. Batch 2 cores were then sandblasted (like batch 1) and the veneering porcelain was applied.
Figure 48. Glass infiltrated alumina core from batch 1.
Figure 49. Glass infiltrated alumina core from batch 2.
Water storage times

While disks were randomly assigned to frequency groups (2 Hz, 10 Hz, 20 Hz), water storage times were not randomized and it happened that disks spent significantly different times in water storage prior to testing (Fig. 36-38). Time constraints did not allow for the storage times of nearly 3 months, as used by Hunter and Kelly (2000). An assumption was made that all storage times should be a minimum of two weeks and that a week or two of additional storage would not alter the failure behavior.

It is not clear from the data that water storage time had an influence, since: (1) within each batch there was no significant difference in storage times for failed versus run-out disks; (2) for disk set where a significant load difference was found (20 Hz being higher than 2 Hz) there was no difference in water storage times among any of the four groups (2 Hz failed and run-out; 20 Hz failed and run-out); and (3), for all batch 2 disks combined (2 Hz, 10 Hz, and 20 Hz), there was no significant difference in water storage times for failed (33.6 days ± 2.2 days) versus run-out disks (28.3 days ± 2.4 days).

However, an influence on failure loads by pre-test water storage times cannot be clearly ruled-out either and deserves further discussion.

Properties of zinc phosphate cement are typically measured as-set and at 24 hours, and it does not appear that further property changes happen on the scale of time used in this project (14 days to 40 days). In a report just published, the strength of zinc phosphate did not change with water storage from 24 days to 150 days (Piwowarczyk and Lauer, 2003).
Water is well known to influence failure of ceramics and by implication was suspected in possibly influencing the ability of the cement to withstand high numbers of low cyclic loads. A key design element of the present protocol was the water access to the cementation surface of the specimen to influence both the cement and the ceramic. Water was transported through the microtubules drilled in the supports (via capillary action or “pumping” driven by cyclic loading) and at the edges of the disk by diffusion (or transport) through the cement. Zinc phosphate is a soluble cement and the water may have affected it. However, nothing appears to have been published regarding the diffusion or transport of water through zinc phosphate cement. In addition, this study did not investigate the integrity of the cement at the end of each loading test, neither was it possible to verify that the water was really able to reach the ceramic through the cement layer. An indirect proof of the cement integrity was that at the time of the de-bonding, all the disks were firmly cemented to their supports and a conspicuous impact delivered to the edge of each disk was necessary to break the bond. Finally, due to the different length of testing at the different frequencies, the specimens loaded at 2Hz were exposed for several hours longer to the “pumping effect” of the cyclic loading (Fig. 50). In a future study, more attention should be paid in establishing a stricter protocol for the total water exposure of each disk (both during the storage and the testing).
Figure 50. Water refill during testing to compensate for evaporation.
7. CONCLUSIONS

Under the conditions of this study, the following conclusions can be made. It has been demonstrated that failure cracks arising from the cementation surface can be produced in a repeatable manner in bi-layered dental ceramic disks. Methods were developed to minimize contact surface stresses in order to suppress the formation of clinically meaningless (regarding bulk fracture) contact surface cracks. Protocol details were established for the fabrication of specimen bases, control of cement layer thickness, removal of cemented discs for examination, and the alignment of specimens for loading. It was established that subsurface cracks, originating in the core ceramic, invariably propagate into the veneer ceramic where they are easily visualized. Coefficients of variation of approximately 10% were achieved with a modest number of specimens (≤ 20) by using a two-phase approach to staircase sensitivity testing: initial larger step size (50 N) to determine approximate mean load; smaller step size (25 N) for testing. It appears that there may have been a small beneficial fatigue effect when testing was performed at 20 Hz versus 2 Hz. However, this fatigue effect was quite small (app. 5%) and it is concluded that future comparative testing can be performed at 20 Hz, greatly speeding data accumulation. It is not known whether water storage time, prior to testing, influenced mean failure loads. However, it is suggested that this either be studied or controlled for in future studies.
8. FUTURE WORK

There are many issues to be investigated, both practical and basic, deriving from this work. Some practical questions relate to the role that different cements may or may not play in failure lifetimes, the comparison of various core-veneer ceramic systems (including those produced in a full crowns configuration instead of a disk), the influence of core-veneer thickness ratios on cyclic failure loads, and the degree to which various dental and dental laboratory processes (e.g. sandblasting) control performance. Questions related to the fatigue effect, the role of bonding, and damage accumulation in the dental cement are more basic but available for study via the protocol developed and used in this research.

Table 3
In-Ceram alumina, batch 1, 10 Hz
less frequent event = failure

<table>
<thead>
<tr>
<th></th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>475</td>
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<td>0</td>
<td>0</td>
</tr>
<tr>
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<td>1</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
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<td>0</td>
</tr>
<tr>
<td>550</td>
<td>3</td>
<td>4</td>
<td>12</td>
<td>36</td>
</tr>
</tbody>
</table>

I = all stress levels experienced by less frequent event (low at top)
II = 0 assigned to lowest, 1 to next, 2 to next etc.
III = number of times the event occurred at each stress level
IV = product of column II x column III
V = product of square of column II x column III

A = sum of column IV
B = sum of column V

d = step size

So = lowest stress for less frequent event

N = total number of less frequent events

Sm (mean strength) = 522.5

\( X = 1.44 \)

if \( X > or = 0.3; \) then use SD1

\[ SD1 = 59.49 \quad CV = 11.4\% \]

if \( X < or = 0.3; \) then use SD 2

\[ SD2 = 13.25 \quad CV = 2.5\% \]

\( note: \) adjust +/- in Sm: (+) if less frequent event is runout;
(-) if failure
Staircase Sensitivity Design Statistics, based on J.A. Collins, "Failure of Materials in Mechanical Design"

<table>
<thead>
<tr>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>0</td>
<td>0</td>
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<tr>
<td>575</td>
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<tr>
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<td>0</td>
</tr>
</tbody>
</table>

Table 4
In-Ceram alumina, batch 2, 10 Hz
less frequent event = runout

I = all stress levels experienced by less frequent event (low at top)
II = 0 assigned to lowest, 1 to next, 2 to next etc.
III = number of times the event occurred at each stress level
IV = product of column II x column III
V = product of square of column II x column III

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>d</th>
<th>So</th>
<th>N</th>
<th>Sm (mean strength)</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
<td>29</td>
<td>25</td>
<td>550</td>
<td>8</td>
<td>603.13</td>
</tr>
</tbody>
</table>

\[ X = 0.984375 \]

if \( X \geq 0.3 \); then use SD1
\[ SD1 = 41.04 \quad CV = 6.8\% \]

if \( X < 0.3 \); then use SD 2
\[ SD2 = 13.25 \quad CV = 2.2\% \]

note: adjust +/- in Sm: (+) if less frequent event is runout;
(-) if failure
Table 4a
In-Ceram alumina, batch 2, 10 Hz
less frequent event = failure

<table>
<thead>
<tr>
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<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
</tr>
</thead>
<tbody>
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<td>0</td>
<td>0</td>
</tr>
<tr>
<td>600</td>
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<td>3</td>
</tr>
<tr>
<td>625</td>
<td>2</td>
<td>2</td>
<td>4</td>
<td>8</td>
</tr>
<tr>
<td>650</td>
<td>3</td>
<td>2</td>
<td>6</td>
<td>18</td>
</tr>
</tbody>
</table>

II = 0 assigned to lowest, 1 to next, 2 to next etc.

III = number of times the event occurred at each stress level

IV = product of column II x column III

V = product of square of column II x column III

A = sum of column IV
B = sum of column V

A = 13
B = 29

Note: adjust +/- in Sm: (†) if less frequent event is runout;
(-) if failure

X = 0.984375

if X > or = 0.3; then use SD1

\[ SD1 = 41.04 \quad CV = 6.8\% \]

if X < or = 0.3; then use SD 2

\[ SD2 = 13.25 \quad CV = 2.2\% \]

Sm (mean strength) = 603.13

<table>
<thead>
<tr>
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<th>III</th>
<th>IV</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>525</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>550</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>575</td>
<td>3</td>
<td>3</td>
<td>9</td>
<td>27</td>
</tr>
<tr>
<td>600</td>
<td>4</td>
<td>1</td>
<td>4</td>
<td>16</td>
</tr>
</tbody>
</table>

\[ \begin{align*}
A &= \text{sum of column IV} \\
B &= \text{sum of column V} \\
d &= \text{step size} \\
So &= \text{lowest stress for less frequent event} \\
N &= \text{total number of less frequent events}
\end{align*} \]

\[ \text{Sm (mean strength)} = 569.64 \]

Note: adjust +/- in Sm: (+) if less frequent event is runout; (-) if failure

\[ X = 1.632653 \]

if \( X > \text{or} = 0.3 \); then use SD1
\[ \text{SD1} = 67.30 \quad \text{CV} = 11.8\% \]

if \( X < \text{or} = 0.3 \); then use SD 2
\[ \text{SD2} = 13.25 \quad \text{CV} = 2.3\% \]

Table 6
In-Ceram alumina, batch 2, 20 Hz
less frequent event = runout

<table>
<thead>
<tr>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>575</td>
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<td>1</td>
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<tr>
<td>600</td>
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<td>2</td>
<td>4</td>
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<tr>
<td>625</td>
<td>3</td>
<td>1</td>
<td>3</td>
<td>9</td>
</tr>
</tbody>
</table>

II = 0 assigned to lowest, 1 to next, 2 to next etc.
III = number of times the event occurred at each stress level
IV = product of column II x column III
V = product of square of column II x column III

A = sum of column IV
B = sum of column V
d = step size
So = lowest stress for less frequent event
N = total number of less frequent events

X = 1.04

if X > or = 0.3; then use SD1
SD1 = 43.29 CV= 7.2%
if X < or = 0.3; then use SD 2
SD2 = 13.25 CV= 2.2%

X = 1.04

if X > or = 0.3; then use SD1
SD1 = 43.29 CV= 7.2%
if X < or = 0.3; then use SD 2
SD2 = 13.25 CV= 2.2%

Sm (mean strength) = 602.50

Note: adjust +/- in Sm: (+) if less frequent event is runout; (-) if failure
Routine to calculate t statistic from means and standard deviations
(Scheffler WC (1979). Statistics for the Biological Sciences,
Reading, MA: Addison-Wesley, pp 89-93).

<table>
<thead>
<tr>
<th>INPUTS</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean A (Xa)</td>
<td>602.50</td>
<td>SDa = 43.29</td>
<td>n(a) = 5</td>
</tr>
<tr>
<td>Mean B (Xb)</td>
<td>569.64</td>
<td>SDb = 67.30</td>
<td>n(b) = 7</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>OUTPUTS</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>variance a (Ssa)</td>
<td>1874.0241</td>
<td></td>
<td></td>
</tr>
<tr>
<td>variance b (SSb)</td>
<td>4529.29</td>
<td></td>
<td></td>
</tr>
<tr>
<td>pooled variance (Sp2)</td>
<td>640.33</td>
<td></td>
<td></td>
</tr>
<tr>
<td>standard error (Sxa-Xb)</td>
<td>14.82</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\[
\text{t} = \frac{602.50 - 569.64}{14.82} \approx 2 \\
\text{df} = 10
\]

Table 7.
2 Hz versus 20 Hz; low n

101
Routine to calculate t statistic from means and standard deviations

**INPUTS**

<table>
<thead>
<tr>
<th>Mean A (Xa)</th>
<th>Mean B (Xb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>602.50</td>
<td>569.64</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>SDa =</th>
<th>SDb =</th>
</tr>
</thead>
<tbody>
<tr>
<td>43.29</td>
<td>67.30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>n(a) =</th>
<th>n(b) =</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
<td>15</td>
</tr>
</tbody>
</table>

**OUTPUTS**

| variance a (Ssa) =         | 1874.0241   |
| variance b (Ssb) =         | 4529.29     |

| pooled variance (Sp2) =    | 246.28      |

| standard error (Sxa-Xb) =  | 5.95        |

| t =                       | 6           |
| df =                      | 26          |

Table 7a
2 Hz versus 20 Hz; high n
9. REFERENCES


