Comparison of Load-to-Fracture Values and Fracture Characterization of Monolithic Zirconia, Lithium Disilicate, and Zirconia-Reinforced Lithium Silicate Glass-Ceramic CAD/CAM Crowns

Thesis

Presented in Partial Fulfillment of the Requirements for the Degree Master of Science in the Graduate School of The Ohio State University

By

Afnan Kashkari

Graduate Program in Dentistry

The Ohio State University

2016

Master’s Examination Committee:

Advisor: Burak Yilmaz, DDS, PhD

Member: William A. Brantley, BS, MS, PhD

Member: William M. Johnston, BS, MS, PhD

Member: Scott R. Schricker, BS, MS, PhD
Abstract

All-ceramic monolithic crowns have been extensively used in prosthodontics in recent years for their excellent advantages compared to traditional metal-based restorations. In the oral environment, ceramic restorations are susceptible to cyclic fatigue failure, mainly due to the presence of cyclic masticatory forces together with the moist environment. The purpose of the study was to compare the integrity of zirconia, lithium disilicate, and zirconia-reinforced lithium silicate monolithic CAD/CAM crowns after being subjected to cyclic loading in a wet condition and then subjected to static loading until fracture. A fiberglass master die replica was used to fabricate (1) zirconia (Zirkonzahn, South Tyrol, Italy), (2) lithium disilicate (LDS, IPS e.max® CAD, Ivoclar Vivadent AG, Schaan / Liechtenstein), and (3) zirconia-reinforced lithium silicate glass ceramic (ZLS), Vita suprinity, VITA Zahnfabrik, Bad Säckingen, Germany) monolithic crowns that were milled using CAD/CAM technology. The crowns were bonded according to manufacturer instructions and subjected to cyclic loading (10,000 and 50,000 cycles) under wet conditions. Representative specimens from each group were examined using SEM. Analysis of the recorded load-to-fracture values was carried out with two-way (ANOVA) followed by the Tukey-Kramer post hoc test ($\alpha=.05$). The mean of load-to-fracture values measured were 3232.27 N for 10,000-cycle LDS, 3510.62 N for 50,000-cycle LDS, 1207.69 N for 10,000-cycle ZLS, 761.20 N for 50,000-cycle ZLS, 4967.29 N for 10,000-
cycle zirconia, and 5041.99 N for 50,000-cycle zirconia. All mean load-to-fracture values were statistically significantly different (p< .05). However, there was no statistical influence of the number of cycles on the load-to-fracture of test crowns (p > .05). Load-to-fracture values of Zirconia and LDS CAD/CAM crowns were above the reported clinical posterior occlusal forces (720 N according to Gibbs et al.) and ZLS load-to-fracture values were closest to the average posterior occlusal force value. Further clinical and laboratory investigation are needed on the use of ZLS ceramic.
Dedication

This thesis is dedicated in memory of my father Abdulahad Kashkari who taught me to work hard for the things that I aspire to achieve. This work is also dedicated to my mother, Mrs. Hayat Zaghabah, my husband Dr. Faris Alshahrani, and my daughters Jana and Mariam, for being constant sources of love, support, and encouragement throughout my life.
Acknowledgments

I would like to thank my advisor Dr. Burak Yilmaz for his guidance and support throughout the study. I would also like to thank my committee member, Dr. William Brantley, in a special way; I express my heartfelt gratefulness for his guidance and support that I believed I learned from the best. Also, I would like to thank my committee members, Dr. William Johnston, for his timely help with the statistics and interpretation of the results presented in this thesis, and Dr. Scott Schricker for his comments and questions that were very beneficial in my completion of the discussion and understanding the problems. Special thanks to Dr. Robert Seghi and Dr. Jeremy Seidt for their unlimited help throughout the laboratory testing.
Vita

2009…………………………………….. Bachelor in Dental Medicine & Surgery

Faculty of Dentistry,

King Abdulaziz University

2012-2014………………………………. Advanced Operative Program, University of

Southern California (USC)

2014-Present…………………………… MS Program in Dental Material Science,

The Ohio State University (OSU)

Fields of Study

Major Field: Dentistry
# Table of Contents

Abstract......................................................................................................................ii

Dedication..................................................................................................................iv

Acknowledgements.....................................................................................................v

Vita..............................................................................................................................vi

List of Tables..............................................................................................................viii

List of Figures............................................................................................................ix

Chapter 1: Introduction..............................................................................................1

Chapter 2: Materials and Methods...........................................................................7

Chapter 3: Results......................................................................................................18

Chapter 4: Discussion...............................................................................................24

Chapter 5: Conclusion...............................................................................................30

References..................................................................................................................31

Appendix A: Load-to-fracture test — load, time, and displacement values ..........36
List of Tables

Table 3.1 Mean and standard deviation for material and number of cycles for the load-to-fracture test………………………………………………………………………………………………………………………19

Table 3.2 ANOVA summary for materials, cycles, and material-cycles interaction……20

Table 3.3 Differences of means – material types – pairwise comparisons (EC = LDS, VS = ZLS, Zir = Zirconia)……………………………………………………………………………………………………20
List of Figures

Figure 2.1 Ivory mandibular first molar tooth.................................................8

Figure 2.2 Complete coverage tooth preparation...........................................8

Figure 2.3 G10 Fiberglass pucks.................................................................9

Figure 2.4 Scanning and designing the crown on the abutment (occlusal view).....10

Figure 2.5 Scanning and designing the crown on the abutment (buccal view)......11

Figure 2.6 Crown nested in the block ........................................................11

Figure 2.7 Simulating the milling cycle showing all process steps .................11

Figure 2.8 Abutment and CAD/CAM-milled ZLS crown ...............................12

Figure 2.9 Used 5% HF etchant.................................................................13

Figure 2.10 Silanes used for zirconia, ZLS, and LDS crowns .......................13

Figure 2.11 The self-adhesive cement used ...............................................14

Figure 2.12 Abutment embedded in Rock Core composite resin .................15

Figure 2.13 Specimen under cyclic loading test .......................................16
Figure 3.1: Means (95% confidence limits) of failure load for all groups (EC = LDS; VS = ZLS; Zir = Zirconia) …………………………………………………...19

Figure 3.2 SEM photographs illustrating various fractographic features in the monolithic crowns ……………………………………………………….21
Chapter 1: Introduction

Computer-aided design (CAD) and computer-aided manufacturing (CAM) technologies have become increasingly popular in dentistry over the past 25 years. The technology, which is used in both the dental laboratory and the dental office, has different applications: inlays, onlays, crowns, veneers, fixed partial dentures, implant abutments, and also full-mouth reconstructions. CAD/CAM technology was developed mainly to address 3 challenges. The first challenge was to provide a restoration for the patient that has adequate strength, especially for posterior teeth. The second challenge was to design restorations with a natural appearance. The third challenge was to make the fabrication procedure easier, faster, and more accurate. In some cases, CAD/CAM technology has the ability to provide patients with same-day restorations.

All-ceramic monolithic crowns have been extensively used in prosthodontics in recent years for their excellent biologic response, superior esthetics, and marginal accuracy comparable to traditional metal-based restorations. Patients demand for metal-free restorations and the success of all ceramic crowns have led to the development and introduction of restorative systems for all-ceramic materials. These systems continue to be evaluated in clinical and laboratory studies for their predictability and long-term success.
In the oral environment, ceramic restorations are susceptible to cyclic fatigue failure, mainly due to the presence of cyclic masticatory forces together with the moist environment. Wiskott et al. defined fatigue failure as the fracture of the material due to progressive brittle cracking under repeated cyclic stresses with intensity below the material normal strength. In the literature and previous studies examining the fracture strength of ceramic crowns, few have taken into consideration the effect of mechanical fatigue. The repetitive load cycling during mastication causes cracks to originate from material flaws in a ceramic material and continue to propagate through its bulk. In the last 30 years, researchers have employed the discipline of fracture mechanics for analyzing the fracture properties of ceramics and predicting their fatigue behavior. Slow crack growth during fatigue is described in terms of a fracture mechanics analysis of ceramic materials. A wet environment, such as present in the oral cavity, accelerates the degradation of dental ceramics, subsequently affecting their mechanical performance. Drummond et al. showed that ceramic materials which were tested after cyclic fatigue in water demonstrated significantly lower strength than non-fatigued specimens. Southan and Jorgensen showed that the ability of dental porcelain to sustain a static load in an aqueous environment, specifically water, decreased as the duration of load application increased. Also, Jones stated that decreasing strength with decreasing stress/strain rate provided further evidence for the detrimental role-played by water. The oral environment seems to have all the factors necessary for fatigue failure to occur in ceramic-based dental prostheses. Water is the primary chemical component of saliva.
According to Denry and Kelly, unalloyed zirconia can assume three crystallographic forms depending on the temperature. At room temperature and upon heating up to 1170°C, the crystal structure is monoclinic, which has reduced mechanical properties. The structure is tetragonal, which has improved mechanical properties, between 1170 and 2370°C, and cubic above 2370°C to the melting point, which has moderate mechanical properties. The excellent mechanical properties of yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP) ceramics can be attributed to the zirconia transformation toughening mechanism that acts to resist crack propagation. Upon cooling and under stress, the Y-TZP tetragonal phase may transform into the monoclinic state, resulting in 3–4% volume expansion, which induces compressive stress in the area of the crack, preventing further crack propagation and increase the resistance to crack growth. The transformation is reversible and begins on cooling at around 950°C. Alloying pure zirconia with stabilizers such as CaO, MgO, Y₂O₃, or CeO₂ allows the stabilization and retention of the tetragonal structure at room temperature. Therefore, the control of the stress-induced $t \rightarrow m$ transformation efficiently arrests crack propagation and leads to high fracture toughness. However, this phenomenon can also change the phase integrity of the material, increasing its susceptibility to aging. As zirconia ages, its strength may decrease due to phase transformation, which was also shown to occur as a result of mechanical fatigue. In order to simulate intraoral environment properly, ceramic restorations should undergo cyclic loading in an aqueous solution during in vitro evaluation.
A zirconia-containing lithium silicate (ZLS) material was recently introduced as a machinable ceramic for CAD/CAM (Suprinity®, Vita). This material is claimed to have mechanical properties comparable with those of lithium disilicate glass-ceramics (L2S).\textsuperscript{16} The technology of this material relies on the addition of 10 wt% zirconium oxide to lithium silicate glass. Multicomponent systems provide the possibility of controlled nucleation and crystal growth, resulting in materials of adjustable microstructure and properties.\textsuperscript{16,17} Kruger et al. and Denry have stated that zirconia acts as nucleating agent but remains in solution in the glassy matrix, with two main consequences: (1) a dual microstructure consisting of very fine lithium metasilicate ($\text{Li}_2\text{SiO}_3$) and lithium disilicate ($\text{Li}_2\text{Si}_2\text{O}_5$) crystals and (2) a glassy matrix containing zirconium oxide in solution.\textsuperscript{16,17}

In addition, Lin et al. studied the structure of lithium disilicate glasses containing 10 wt% ZrO$_2$ using Raman scattering, and found that ZrO$_2$ acts like a glass network former, promoting amorphous-phase separation.\textsuperscript{18} Apel et al. added up to 4 wt% ZrO$_2$ to a multicomponent lithium disilicate glass with minor constituents of Al$_2$O$_3$, K$_2$O, and P$_2$O$_5$ to improve the mechanical strength and translucency of the glass-ceramics.\textsuperscript{19} They found in their study that ZrO$_2$ decreased the fractions of both primary lithium metasilicate and secondary lithium disilicate crystals. They assumed that there is an increase in the
viscosity of the ZrO$_2$-bearing glasses which is responsible for the smaller crystal growth rates.\textsuperscript{19} With respect to applications, lithium disilicate glass-ceramics containing ZrO$_2$ concentrations up to 10 wt\%, and sometimes 20 wt\%, are considered advantageous.\textsuperscript{20,21} However, the influence of ZrO$_2$ on the nucleation rate, crystal number density, and the corresponding viscosity has not been studied.

Denry and Kelly explained the two-stage microstructure of ZLS.\textsuperscript{22} The first stage is the pre-crystallized, “green” stage that is easy to machine, which contains only lithium metasilicate crystals in the structure. After a short heat treatment at 840°C for 8 min, the final crystallization stage is obtained, leading to the dual lithium silicate microstructure [consisting of very fine lithium metasilicate (Li$_2$SiO$_3$) and lithium disilicate (Li$_2$Si$_2$O$_5$) crystals]. It can be concluded that the main difference between ZLS and L2S glass-ceramics is in their final stage of crystallization, residing in the nature of the crystalline phases. ZLS has two types of crystals (lithium metasilicate and lithium disilicate), while L2S has lithium disilicate crystals only in the final stage of crystallization.\textsuperscript{22}

For the multicomponent lithium disilicate glasses, the crystallization sequence: lithium metasilicate (around 793 K), lithium disilicate (around 793 K and 1023 K), and lithium orthophosphate (around 1103 K) upon heating, was reported in previous studies.\textsuperscript{17,23,24}
The development of zirconia-containing lithium silicate glass-ceramics may offer more reliable restorations compared to zirconia ceramics. However, further investigations and research are needed. 22

The purpose of this study was to compare the integrity of zirconia, lithium disilicate, and zirconia-reinforced lithium silicate monolithic CAD/CAM crowns after being subjected to cyclic loading under wet conditions and load-to-fracture values after static loading.

The null hypothesis was that there would be no difference in the integrity and load-to-fracture values among the three restorative materials when subjected to cyclic loading. Also, there would be no difference in load-to-fracture values between 10,000 and 50,000 cycles of fatigue before testing.
Chapter 2: Materials and Methods

*Master Die Fabrication*

A mandibular ivory first molar tooth (Fig. 2.1) was used to produce a master die replica. The tooth was duplicated using a putty impression material (Extrude XP, Kerr) to produce a silicone key representing the outer form of the ivory tooth before preparation. Then the tooth was prepared for a complete coverage crown (1.5 mm occlusal reduction and 1 mm circumferential reduction) (Fig. 2.2). The prepared tooth was scanned (3shape D800 scanner, 3Shape A/S, Holmens Kanal 7, 4, 1060 Copenhagen K Denmark) to mill fiberglass replicas (G10, McMaster-Carr, Aurora, OH) (Fig. 2.3), using a CAD/CAM system (Wieland Dental Zenotech mini milling machine, Wieland Dental + Technik GmbH & Co. KG Schwenninger Straße 13, 75179 Pforzheim, Germany). The milled replicas were used as master dies (n=18).
Fig. 2.1 Ivory mandibular first molar tooth

Fig. 2.2 Complete coverage tooth preparation
Crown Fabrication

A complete contour wax-up was accomplished by injecting melted wax through the perforated silicone key. The wax-up was checked in the cyclic loading machine to verify the contact between the crown and the load application indenter, and to make an indentation in the wax crown for the indenter to fit properly. The wax-up on the master die was mounted on the scanning table and scanned, using a laboratory scanner (S600 ARTI, Zirkonzahn, South Tyrol, Italy) to fabricate crowns for all groups (Fig. 2.4) and (Fig. 2.5).

The fabricated monolithic crowns were divided into three groups of 6 specimens each. Group A consisted of ZLS (Vita Suprinity, VITA Zahnfabrik, Bad Säckingen, Germany) crowns; group B consisted of LDS (IPS e.max® CAD, Ivoclar Vivadent AG, Schaan/Liechtenstein) crowns, and group C consisted of zirconia (Prettau® Zirconia, Zirkonzahn, South Tyrol, Italy). The digital information was sent electronically to the CAM unit (Zirkonzahn Milling unit M1, Zirkonzahn, South Tyrol, Italy) to mill the crowns. See
The milled crowns were seated on their corresponding abutments to evaluate the marginal fit. Any crowns with a marginal gap exceeding the thickness of the sharp tip of an explorer (39 µm) was planned to be discarded; however, none failed to meet this criterion. The crowns were sintered in a furnace, and then polished to a final high luster with a diamond-impregnated polishing instrument (Dialite ZR Extra-oral; Brasseler). The crowns were then glazed and crystallized in an oven (Programat, Ivoclar) under vacuum according to the manufacturer instructions for each material. (Fig. 2.8). The crown thicknesses were checked occlusally and circumferentially.
Fig. 2.5 Scanning and designing the crown on the abutment (buccal view)

Fig. 2.6 Crown nested in the block

Fig. 2.7 Simulating the milling cycle showing all process steps
Crown Surface Treatments

The manufacturer instructions for each crown material were followed. For standardization, the same operator performed the bonding procedure. The ZLS and LDS groups were etched with 5% hydrofluoric acid (IPS ceramic etchant gel, Ivoclar Vivadent AG, Schaan / Liechtenstein) for 20 s. The hydrofluoric acid was rinsed off with a water spray. The crowns were cleaned in an ultrasonic bath (1 – 3 min in 98% alcohol). The silane [Monobond® Plus (Ivoclar Vivadent AG, Schaan / Liechtenstein)] for LDS and ZLS crowns was applied, allowed to react for one minute, and then dispersed by applying air for 5 s. This procedure enabled a very thin silane coat to be obtained.

The zirconia crowns were air-abraded with alumina powder having a mean particle size of 110 µm for 10 s at a pressure of 3.5 bar and a distance of 10 mm, followed by a steam blast, and dried with compressed air. Clearfil ceramic primer (Kuraray, Tokyo, Japan) was applied, allowed to react for 1 min, and then dispersed by applying air for 5 s. This procedure also enabled a very thin silane coat to be obtained (Fig. 2.9) and (Fig. 2.10).
Fig. 2.9 Used 5% HF etchant

Fig. 2.10 Silanes used for zirconia, ZLS, and LDS crowns

*Crown Cementation*

A self-adhesive cement [RelyX Unicem (3M ESPE, St Paul, MN, USA)] was used for cementation of the ZLS crowns and zirconia crowns, and Speed CEM (Ivoclar Vivadent AG, Schaan / Liechtenstein) was used for cementation of the LDS crowns (Fig. 2.11).
The test crowns were cemented on their corresponding air-abraded abutments using a 5-kg weight applied on the occlusal surface during setting to ensure complete seating. The load was removed after 10 min, and the excess cement was carefully removed from the margins with disposable brushes.

All of the margins were light-polymerized for 40 s within a distance of 0.5 mm from the specimen with a curing unit (Optilux 500, Kerr Dental Products, USA; light intensity ~500 W/m²), which was monitored with a radiometer before each cementation. Each crown was placed in the middle of a metallic ring (3 mm below the crown margins) and embedded in a dual-polymerized composite resin core build-up material (Rock Core, Danville Materials, San Ramon, CA) (Fig. 2.12). The specimens were stored in distilled water until cyclic loading was performed.
Fatigue Test and Loading Procedures

Specimens were then subjected to cyclic loading, simulating mouth motion under wet conditions (distilled water) at 37°C. Three specimens from each group were loaded for 10,000 cycles, and the other three specimens were loaded for 50,000 cycles with a profile of 250 N maximum load at 1,000 N/s rate and 2.0 Hz frequency. Loading was performed using a mechanical testing machine (Model 812.21, MTS, Eden Prairie, MN) with a steel ball (5 mm in diameter) contacting the test crown at the indentation. In order to avoid force peaks, a piece of tin foil was placed between the crown and the indenter. After the cyclic loading was completed, the crowns from all groups were inspected using a low-power stereomicroscope to identify the presence of any cracks or fractures. Then, the specimens were subjected to a load-to-fracture test at a 1.0 mm/min loading rate (Fig. 2.13).
Representative specimens from each group were examined with a scanning electron microscope (Quanta 200, FEI, Hillsboro, OR) to observe any cracks and cohesive failures.

**Statistical Analysis**

The load-to-fracture data were summarized using means, standard deviations and 95% confidence intervals, and analyzed with a two-way ANOVA using the method of maximum likelihood estimation\(^25\) (SAS MIXED Procedure, SAS (R) Proprietary Software 9.3, SAS Institute, Cary, NC) in order to account for any violations of normality or inequality of variances. The two factors were the material and number of cycles. The interaction of these main factors was included in this statistical model. Tukey testing was
subsequently applied to pairwise comparisons in order to resolve any found statistically significant effect with greater than one degree of freedom ($\alpha = .05$).
Chapter 3: Results

None of the zirconia or LDS specimens fractured or cracked during the cyclic loading. Four out of six ZLS specimens cracked the day after bonding, before starting the cyclic loading test. The cracked specimens were replaced with new ZLS specimens that were milled, fired, glazed, and bonded according to the manufacturer instructions. Three of the new ZLS specimens cracked during cyclic loading: 1 crown during 10,000 cycles and 2 crowns during 50,000 cycles. Therefore, the data for specimens that cracked during cycling loading were treated as zeros for analysis of the load-to-fracture test results.

The results of the load-to-fracture test for all of the groups are presented in Table 3.1. All mean values of the load-to-fracture were statistically significant among groups, which means that the material had a significant influence on sustaining the fracture load (p < .05). There was also no significant influence found on the number of cycles (p > .05). After 10,000 cycles and 50,000 cycles of loading for the zirconia and LDS groups, the crowns displayed no crack formation or bulk fracture. Crack formation was found on the occlusal surface of 1 crown after 10,000 cycles and 2 crowns after 50,000 cycles in the ZLS group. The initial crack was observed in the monolithic ZLS material at the end of the cyclic loading; therefore, the crack origin could not be determined. The interaction between the materials and number of cycles was not statistically significant (p > .05); see
Table 3.1 Mean and standard deviation for material and number of cycles for the load-to-fracture test.

<table>
<thead>
<tr>
<th>Material</th>
<th>e.max CAD (LDS)</th>
<th>Vita Suprinity (ZLS)</th>
<th>Zirconia</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Cycles</td>
<td>10,000</td>
<td>10,000</td>
<td>10,000</td>
</tr>
<tr>
<td></td>
<td>50,000</td>
<td>50,000</td>
<td>50,000</td>
</tr>
<tr>
<td>Mean (N)</td>
<td>3232</td>
<td>1207</td>
<td>4967</td>
</tr>
<tr>
<td></td>
<td>3510</td>
<td>761</td>
<td>5041</td>
</tr>
<tr>
<td>SD (N)</td>
<td>404</td>
<td>1046</td>
<td>235</td>
</tr>
<tr>
<td></td>
<td>202</td>
<td>1318</td>
<td>194</td>
</tr>
</tbody>
</table>

Table 3.2 and Table 3.3.

Figure 3.1: Means (95% confidence limits) of failure load for all groups

(EC = LDS; VS = ZLS; Zir = Zirconia)
<table>
<thead>
<tr>
<th>Effect</th>
<th>Num</th>
<th>Den</th>
<th>F Value</th>
<th>Pr &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>2</td>
<td>12</td>
<td>70.52</td>
<td>&lt;.0001</td>
</tr>
<tr>
<td>Cycles</td>
<td>1</td>
<td>12</td>
<td>0.01</td>
<td>0.9126</td>
</tr>
<tr>
<td>Material*Cycle</td>
<td>2</td>
<td>12</td>
<td>0.60</td>
<td>0.5631</td>
</tr>
</tbody>
</table>

Table 3.2 ANOVA summary for materials, cycles, and material-cycles interaction.

<table>
<thead>
<tr>
<th>Material</th>
<th>Material</th>
<th>Estimate</th>
<th>Standard</th>
<th>DF</th>
<th>Adjustment</th>
<th>Adj P</th>
</tr>
</thead>
<tbody>
<tr>
<td>EC</td>
<td>VS</td>
<td>2386.99</td>
<td>340.49</td>
<td>12</td>
<td>Tukey</td>
<td>&lt;.0001</td>
</tr>
<tr>
<td>EC</td>
<td>Zir</td>
<td>-1633.20</td>
<td>340.49</td>
<td>12</td>
<td>Tukey</td>
<td>0.0012</td>
</tr>
<tr>
<td>VS</td>
<td>Zir</td>
<td>-4020.19</td>
<td>340.49</td>
<td>12</td>
<td>Tukey</td>
<td>&lt;.0001</td>
</tr>
</tbody>
</table>

Table 3.3 Differences of means – material types – pairwise comparisons

(EC = LDS, VS = ZLS, Zir = Zirconia)
Fracture Characterization and SEM

Representative images from the scanning electron microscope (Quanta 200) of fractured pieces of the crowns were used to verify the fracture patterns and the orientation of crystalline grains in the specimens (Fig 3.2). The crack origin in the ceramic crowns was located at the point of contact of the indenter during cyclic loading. In this zone, radiating cracks and hackle features were observed, which indicate the direction of crack propagation that penetrated the entire layer of the crown. Crack propagation started from the occlusal surface of the crowns and propagated towards the interface between the crown and cement, and eventually resulted in bulk fracture. For the zirconia and ZLS SEM images, voids were detected on the inner surface of the crowns. Therefore, it is reasonable to assume that the origins of the cracks were related to these defects.

Fig 3.2 SEM photographs illustrating various fractographic features in the monolithic crowns.

Continued
Fig 3.2 Continued

Continued
Fig. 3.2 SEM photographs illustrating various fractographic features in the monolithic crowns.

(A) Fractured zirconia crown. Voids were detected in the inner surface of the crown. (B) Detailed image of fractured zirconia crown shows the crystal orientation. (C) Image of fractured zirconia crown shows radiating cracks and hackle features, which indicate the direction of crack propagation, combined with voids. (D) Image of fractured LDS crown shows the crystal orientation. (E) Image of fractured LDS crown shows radiating cracks and hackle features. (F) Image of fractured ZLS crown. Large voids were detected on the inner surface of the crown. (G) Detailed image of fractured ZLS crown. (H) Image of fractured ZLS crown shows radiating cracks and hackle features.
Chapter 4: Discussion

The load-to-fracture values amongst the zirconia, lithium disilicate, and zirconia-reinforced lithium silicate monolithic crowns were significantly different \((p < .05)\). The null hypothesis was rejected because cracks were observed with only ZLS crowns. Even though the ZLS group had three specimens only, number of specimens did not have a negative impact on the result obtained because the difference in load-to-fracture values for the three different materials was statistically significant.

In the published literature, different load-to-fracture values have been presented. Altamimi et al. found different results compared to the present study regarding the monolithic LDS load-to-fracture values.\(^3\) In that study it was stated that heat-pressed monolithic LDS crowns showed a higher load-to-fracture value \((1,360\; \text{N})\) compared to bi-layered zirconia/fluorapatite crowns after being subjected to 100,000 loading cycles. Baladhandayutham et al. also compared the load-to-fracture of monolithic and bi-layered LDS and zirconia crowns having clinically relevant thickness after cyclic loading.\(^8\) These investigators found that the monolithic LDS crowns with 1.5 mm occlusal thickness had mean load-to-fracture value of 2027 N compared to 1465N and 1669N values for monolithic LDA crowns with 1.2 mm occlusal thickness and monolithic zirconia crowns with 0.6 mm occlusal thickness, respectively. These values were lower than the values found in the present study.
Similar to this study regarding zirconia restorations, Nakamura et al. studied the kinetics of low-temperature degradation and cyclic loading of monolithic zirconia.\textsuperscript{26} The mean load-to-fracture value was 5683 N before autoclaving. Cyclic loading did not significantly affect the load-to-fracture values of the crowns.

The load-to-fracture values of all materials used in the current study and in the aforementioned previous studies were still sufficient to withstand the loading conditions and the average bite force in the molar region (around 720 N according to Gibbs et al.\textsuperscript{27}). The maximum bite force can vary greatly. It can exceed 800 N in bruxer patients, as reported by Waltimo.\textsuperscript{28} It was found that the mean maximum bite force in the molar region for men was 847 N, and for women 597 N. Moreover, Cosme et al.\textsuperscript{29} when studying the bruxism and voluntary maximal bite force in young dentate adults, found that the mean maximum bite force was 806 N for bruxers. In the current study, for the 50,000-cycle ZLS specimens, the mean load-to-fracture value was lower than some reported maximum bite forces for bruxers. This should be taken into consideration when selecting the proper material for treating patients with severe parafunctional habits.

Self-adhesive cements have become very popular among dentists and been increasingly used during the past few years. The complicated procedure of the conventional multi-step luting agent (etching, priming, and bonding), and the application of the resin cement is highly technique-sensitive. The benefit of self-adhesive cement materials lies in the ability to bond without any type of pre-treatment.\textsuperscript{30,31} Self-adhesive cements are commonly composed of methacrylate monomers with carboxylic acid groups or
phosphoric acid groups. However, the polymerization reaction can be affected by the individual composition of each product, such as the content of inorganic particles and the type of resin monomer, and by extrinsic factors, such as the shade and thickness of the indirect restoration, polymerization lights, and temperature, as well as by the amount of light energy received.\textsuperscript{32-42} The shrinkage strain of RelyX Unicem at 23°C for the self-polymerization and dual-polymerization modes has been tested by Arrais et al.\textsuperscript{42} Their results were 4.1 ± 0.03% compared to 3.7 ± 0.1% for RelyX Unicem dual-polymerized in the study by Aguiar et al.\textsuperscript{43} In self-polymerization mode, there was no difference in the shrinkage strain, being 1.8% in both studies. The bonded disk method was used for shrinkage strain measurements in both studies, and the results seem to be more reproducible when using this method.

Temperature is another influencing factor on polymerization shrinkage strain that was standardized for all the samples during bonding procedure. Temperature increase had an effect on final shrinkage for all materials tested, including Speed Cem cement, except RelyX Unicem in the Aguiar et al. study. Another previous study had concluded that shrinkage strain rate of a light-polymerizing composite resin increases with increasing temperature.\textsuperscript{44} Elhejazi also confirmed this finding that a constant light intensity at a higher temperature produces higher polymerization shrinkage.\textsuperscript{45} Corresponding results were found in the present study for dual-curing materials. It could be possible that the amount of stress generated during polymerization shrinkage of the cement can cause cracks in the crowns. Results of these previous studies may explain the cracks observed with the ZLS crowns during bonding in current study.
The degree of conversion of resin materials is based on the cross-linking of monomer units to form long chains (polymers). Uneven curing has direct impact on physical and mechanical properties for the composite resins and resin luting agents.\textsuperscript{46,47,48,49} In the clinical scenario, total absence or attenuation of light exposure at the apical region and deep interproximal areas, or even the distance from the light source, may impair the long-term success of restorations. Aguiar et al. studied the effect of photopolymerization on the degree of conversion for 4 dual-polymerized resin cements, 20 minutes after mixing, and its effects on the mechanical properties of biaxial flexural strength and elastic modulus. The degree of conversion for each resin cement after 5, 10, and 20 minutes was evaluated. When subjected to direct light exposure, all resin cements showed the highest immediate degree of conversion values. The results also showed that the autopolymerized resin cements did not reach the highest degree of conversion compared to other polymerization conditions at 20-minute dwell time. In addition, when the resin cements were photoactivated, the flexural modulus had significantly higher means in comparison with the autopolymerizing mode for all materials used in the study.\textsuperscript{43}

In the present study, one of the ZLS crowns was luted using the autopolymerization mode. At the next day of bonding before testing, cracks had formed on the occlusal surface, and this crown was excluded from the study. The remaining crowns that were included in the study were bonded according to manufacturer instructions, using photocuring from each surface for 40 seconds within 0.5 mm distance.

The use of natural tooth abutments during testing may simulate the clinical scenario
However, natural teeth vary in size, form, and quality, and potentially would be more difficult for standardizing the tooth preparation. Investigators are using various abutment materials that are easy to produce and standardize; such as epoxy resin or acrylic resin. The supporting die structures should have an elastic modulus similar to teeth to achieve results that may be seen in clinical conditions. The selection of abutment material is vital because the supporting material can play a significant role on the fracture strength of loaded restorations. In the literature, it has been confirmed that the fracture strength of an all-ceramic crown depends on the elastic modulus of the abutment material; as the elastic modulus increases, fracture strength increases. Different abutment materials have different elastic modulus values: enamel 82.5 GPa, dentin (5.2 – 23 GPa), composite resins (8 – 16.5 GPa), G-10 (21 GPa), zirconia (210 GPa), filled epoxy (11.8 GPa), stainless steel (200 GPa), and brass (100 GPa). In this study, a material (flame-retardant multi-purpose Garolite (G-10/FR4) from McMaster-Carr) which has an elastic modulus close to dentin and higher than composite resin was used. This material is a composite composed of woven fiberglass cloth with an epoxy resin binder that is flame-resistant.

During cyclic loading, forces are transferred through the ceramic crown to the underlying cement layer and the core material, inducing stresses and deformation in these structures. Yucel et al. studied the influence of the supporting die structures on the fracture strength of all-ceramic materials, bonding zirconia cores on dentin and epoxy resin dies which showed lower fracture strength values than brass and steel dies. Usually, metal dies are very rigid and have a higher elastic modulus than dentin and epoxy resin.
Under loading, the metal dies deform less, resulting in a lower shear stress at the inner crown surface and higher fracture resistance than what can be seen for a core with a smaller elastic modulus, like dentin.

The result of this study should be interpreted with some caution, considering that fiberglass abutments were used instead of natural teeth and distilled water was used instead of saliva. Future clinical studies of the effect of cyclic (functional) loading on CAD/CAM monolithic crowns are needed to corroborate the result of this study.
Chapter 5: Conclusion

Within the limitations of this study, the following conclusions can be drawn:

1) All mean values were statistically significant within the two loading cycle groups, which means that the material had a significant influence on the load-to-fracture (p < .0001).

2) The number of loading cycles was not found to affect the subsequent load-to-fracture values.

3) The interaction between the materials and number of loading cycles was not statistically significant for the load-to-fracture test (p = .5631).

4) The load-to-fracture values for all crowns were above the reported average posterior occlusal bite force. However, the load-to-fracture for the ZLS crowns was closer to the average occlusal bite force, compared to the zirconia and LDS crowns.
References


Appendix A: Load-to-fracture test — load, time, and displacement values

<table>
<thead>
<tr>
<th>Test #</th>
<th>Load-to-fracture value (N)</th>
<th>Load-to-fracture Time (s)</th>
<th>Load-to-fracture Displacement (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>mono emax 1, 10000</td>
<td>-3092.669537</td>
<td>36.038414</td>
<td>-0.582262996</td>
</tr>
<tr>
<td>mono emax 2, 10000</td>
<td>-3687.704139</td>
<td>49.051109</td>
<td>-0.806995592</td>
</tr>
<tr>
<td>mono emax3,10000</td>
<td>-2916.425036</td>
<td>34.657066</td>
<td>-0.558427128</td>
</tr>
<tr>
<td>mono emax4, 50000</td>
<td>-3621.402729</td>
<td>25.748373</td>
<td>-0.405200104</td>
</tr>
<tr>
<td>mono emax5, 50000</td>
<td>-3277.306275</td>
<td>47.789879</td>
<td>-0.772945876</td>
</tr>
<tr>
<td>mono emax6, 50000</td>
<td>-3633.15235</td>
<td>37.039391</td>
<td>-0.595882476</td>
</tr>
<tr>
<td>mono zir1,10000</td>
<td>-5092.62347</td>
<td>59.361168</td>
<td>-0.93638751</td>
</tr>
<tr>
<td>mono zir2, 10000</td>
<td>-4695.654387</td>
<td>36.138512</td>
<td>-0.578857364</td>
</tr>
<tr>
<td>mono zir3, 10000</td>
<td>-5113.604842</td>
<td>45.527672</td>
<td>-0.725274394</td>
</tr>
<tr>
<td>mono zir4, 50000</td>
<td>-5223.547977</td>
<td>63.685387</td>
<td>-0.806995592</td>
</tr>
<tr>
<td>mono zir5, 50000</td>
<td>-4836.649668</td>
<td>41.423504</td>
<td>-0.670793172</td>
</tr>
<tr>
<td>mono zir6, 50000</td>
<td>-5065.767332</td>
<td>52.664391</td>
<td>-0.841046832</td>
</tr>
<tr>
<td>mono sup1,10000</td>
<td>-1790.978661</td>
<td>27.519531</td>
<td>-0.439251344</td>
</tr>
<tr>
<td>mono sup3,10000</td>
<td>-1832.10247</td>
<td>40.943523</td>
<td>-0.663983432</td>
</tr>
<tr>
<td>mono sup4, 50000</td>
<td>-2283.623698</td>
<td>27.59017</td>
<td>-0.446061338</td>
</tr>
</tbody>
</table>