EFFECT OF COOLING RATE AND THERMAL EXPANSION MISMATCH ON THE APPARENT INTERFACIAL TOUGHNESS OF CORE-VENEER LITHIUM-DISILICATE DENTAL CERAMICS

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ABSTRACT

With sustained interest in esthetic dental restorations, research in all-ceramic technology has led to continued development and improvement. However, the nature of the adherence between a core ceramic and its veneer has proven difficult to measure through traditional methods. Indentation techniques have been proposed to qualitatively evaluate the coating adhesion and apparent interfacial toughness between brittle bilayers. The purpose of the present study is to evaluate the effectiveness of a Vickers indentation technique to assess apparent interfacial toughness of core-veneer dental ceramic bilayers subjected to various cooling rates and exhibiting various thermal expansion mismatches.

A core material of lithium disilicate-based pressable ceramic was veneered with experimental ceramic of high, low or matched coefficient of thermal expansion. The High, Low and Matched groups were sorted into 3 subgroups and subjected to fast, medium or slow cooling rates. Vickers indentation along the interface was conducted to induce interface cracking for each group (n=30). The mean apparent interfacial toughness was then calculated and analyzed statistically with one-way ANOVA and regression analysis.

The apparent interfacial toughness values ranged from 1.21-1.70 MPa·m$^{0.5}$. Apparent interfacial toughness was affected by both cooling rate and coefficient of
thermal expansion mismatch. A Fast cooling rate led to significantly higher apparent interfacial toughness than Medium or Slow cooling rates. A Matched CTE led to increased apparent interfacial toughness, while a High CTE mismatch led to significantly lowered toughness. The Fast cooled-Matched CTE group was significantly higher than the other groups ($p \leq 0.001$), except for the Fast cooled-Low CTE group ($p=0.355$). However, the cooling rate had a greater effect on the apparent interfacial toughness than thermal expansion mismatch.

This Vickers indentation test was an effective method for determining statistically significant differences in apparent interfacial toughness values of bilayered dental ceramics.
I would like to dedicate this work to my family.

Your unconditional love and support always made my day!
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# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abstract</td>
<td>ii</td>
</tr>
<tr>
<td>Dedication</td>
<td>iv</td>
</tr>
<tr>
<td>Acknowledgments</td>
<td>v</td>
</tr>
<tr>
<td>Vita</td>
<td>vi</td>
</tr>
<tr>
<td>List of Tables</td>
<td>ix</td>
</tr>
<tr>
<td>List of Figures</td>
<td>x</td>
</tr>
</tbody>
</table>

## Chapters:

1. Introduction..........................1
2. Materials and Methods....................7
   2.1 Specimen Preparation..................7
   2.2 Coefficient of Thermal Expansion.........11
   2.3 Hardness Testing........................11
   2.4 Density................................11
   2.5 Modulus of Elasticity..................12
2.6 Apparent Interfacial Toughness .................................................. 12
2.7 Residual Stress ........................................................................ 15
2.8 Statistical Analysis .................................................................. 15

3. Results ..................................................................................... 16
  3.1 Coefficient of Thermal Expansion .......................................... 16
  3.2 Vickers Hardness ................................................................... 20
  3.3 Density .................................................................................. 21
  3.4 Elastic Constants .................................................................... 21
  3.5 Critical Load Determinations (Pc) ......................................... 22
  3.6 Apparent Interfacial Toughness ............................................. 37
  3.7 Residual Stress Determinations ............................................ 38
  3.8 Statistical Regression Analysis ............................................. 39

4. Discussion ................................................................................ 45

5. Conclusions ............................................................................. 53

References .................................................................................... 54
# List of Tables

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Mean values for coefficient of thermal expansion, hardness, and density (±SD)</td>
<td>17</td>
</tr>
<tr>
<td>2.</td>
<td>Mean values for Young’s modulus and Poisson’s ratio (±SD)</td>
<td>22</td>
</tr>
<tr>
<td>3.</td>
<td>Apparent interfacial toughness calculation, fast cooling group</td>
<td>24</td>
</tr>
<tr>
<td>4.</td>
<td>Apparent interfacial toughness calculation, medium cooling group</td>
<td>28</td>
</tr>
<tr>
<td>5.</td>
<td>Apparent interfacial toughness calculation, slow cooling group</td>
<td>33</td>
</tr>
<tr>
<td>6.</td>
<td>Mean apparent interfacial toughness values (MPa•m&lt;sup&gt;0.5&lt;/sup&gt;)</td>
<td>37</td>
</tr>
<tr>
<td>7.</td>
<td>Calculated residual stresses of the veneer and core ceramics for each specimen (MPa)</td>
<td>38</td>
</tr>
<tr>
<td>8.</td>
<td>Summary of fit for cooling rate and CTE mismatch ANOVA</td>
<td>43</td>
</tr>
<tr>
<td>9.</td>
<td>Analysis of variance</td>
<td>43</td>
</tr>
<tr>
<td>10.</td>
<td>Prediction formula for apparent interfacial toughness by variable</td>
<td>43</td>
</tr>
</tbody>
</table>
### LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Fast, medium and slow cooling curves</td>
<td>10</td>
</tr>
<tr>
<td>2.</td>
<td>Interface indentation technique, reproduced from Chicot, et al.</td>
<td>13</td>
</tr>
<tr>
<td>3.</td>
<td>Percent linear change for the high veneer group compared to the core ceramic</td>
<td>18</td>
</tr>
<tr>
<td>4.</td>
<td>Percent linear change for the matched veneer group compared to the core ceramic</td>
<td>19</td>
</tr>
<tr>
<td>5.</td>
<td>Percent linear change for the low veneer group compared to the core ceramic</td>
<td>20</td>
</tr>
<tr>
<td>6.</td>
<td>Critical load determination of fast cooling groups (Arrows denote $P_C$)</td>
<td>23</td>
</tr>
<tr>
<td>7.</td>
<td>Interfacial Vickers indentations below, at and above the critical load for the fast cooling, high CTE group (1000x magnification)</td>
<td>25</td>
</tr>
<tr>
<td>8.</td>
<td>Interfacial Vickers indentations below, at and above the critical load for the fast cooling, low CTE group (1000x magnification)</td>
<td>26</td>
</tr>
<tr>
<td>9.</td>
<td>Interfacial Vickers indentations below, at and above the critical load for the fast cooling, matched CTE group (1000x magnification)</td>
<td>27</td>
</tr>
<tr>
<td>10.</td>
<td>Critical load determination of medium cooling groups (Arrows denote $P_C$)</td>
<td>27</td>
</tr>
</tbody>
</table>

x
11. Interfacial Vickers indentations below, at and above the critical load for the medium cooling, high CTE group (1000x magnification).........29
12. Interfacial Vickers indentations below, at and above the critical load for the medium cooling, low CTE group (1000x magnification).........30
13. Interfacial Vickers indentations below, at and above the critical load for the medium cooling, matched CTE group (1000x magnification).........31
14. Critical load determination of slow cooling group (Arrows denote $P_C$)..............32
15. Interfacial Vickers indentations below, at and above the critical load for the slow cooling, high CTE group (1000x magnification)..............34
16. Interfacial Vickers indentations below, at and above the critical load for the slow cooling, low CTE group (1000x magnification)..............35
17. Interfacial Vickers indentations below, at and above the critical load for the slow cooling, matched CTE group (1000x magnification)..............36
18. Influence of cooling rate on apparent interfacial toughness (MPa•m$^{0.5}$).........41
19. Influence of CTE mismatch on apparent interfacial toughness (MPa•m$^{0.5}$).......42
20. Predicted apparent interfacial toughness as a function of observed apparent interfacial toughness, using the equation in Table 14.................................44
CHAPTER 1

INTRODUCTION

The complete veneer gold crown was first developed in 1849 by Beers\(^1\). This type of crown relied on a gold foil substructure, covered with gold solder. It was the first dental restoration that provided encirclement of a tooth. The technique for fabricating these crowns was dramatically improved in 1907 when Taggart adapted the lost wax casting technique to dental applications\(^2\). Gold crowns have many applications, but their use in esthetic situations is limited, therefore many attempts have been made to develop more esthetic crowns. The earliest attempts to fabricate more esthetic crowns occurred in the early 1900’s when metal crowns with bonded porcelain facings were made. These early crowns were subject to fracture in the porcelain layer due to mismatched thermal expansion properties between the metal and the porcelain. It was not until 1962, that Weinstein\(^3\) refined a technique for controlling the coefficient of thermal expansion for feldspathic dental porcelain in order to match that of the metal substructure. Weinstein was able to achieve this by altering the composition of the porcelain. To the feldspathic glass, higher thermal expansion leucite was added to achieve the desired thermal expansion compatibility with the metal substructure. This provided better strength and predictability for metal-ceramic crowns, resulting in long term clinical durability. Since
this time, metal-ceramic crowns have been the standard of care for complete coverage
fixed prosthetic restorations in all areas of the mouth. The main disadvantage of these
types of restorations has been a compromise in esthetics. This is caused by limited
translucency of the veneering porcelain due to the opacity created by the metal
substructure.

Metal-free all-ceramic crowns were first introduced in 1886 by Land when he
described a porcelain jacket crown⁴. Although these types of crowns were very esthetic,
they suffered in terms of strength. The potential to match the esthetics of the natural
dentition has resulted in continued research and development into this field to improve
the strength characteristics. An improvement on the porcelain jacket crown came with
the development of the Dicor® crown⁵. The composition of these crowns consisted of
45% mica crystalline phase in 55% glassy matrix. The refractive index of the mica
crystals matched that of the matrix, accounting for the good translucency and esthetics
provided by the Dicor® crowns. However, the fracture toughness of this material was
relatively low, which led to their frequent failure. To counter this drawback, alumina-
reinforced ceramics have been developed with higher crystalline content and increased
strength properties. As explained previously, the increased strength is usually associated
with an increase in opacity.

The evolution of the all-ceramic crown has reached a compromise where an
opaque, high strength core of ceramic is combined with an esthetic, translucent veneering
porcelain⁶. In the late 1990’s, Ivoclar introduced the Empress (Empress®, Ivoclar,
Amherst, NY) system, which made use of a processing technology known as heat-
pressing. Since this development, many other manufacturers have introduced similar
heat-pressed products. With this technology, ceramic ingots are subjected to simultaneous applications of pressure and temperatures up to 1100°C. The viscosity of the ceramic ingot at these temperatures becomes low enough to allow pressing in a refractory mold\textsuperscript{7}. With minimal additional equipment, this process is easily achieved in a modern dental laboratory. This technique is very similar to the traditional metal casting method of the lost wax technique. The first generation of the heat-pressed ceramics contained about 45\% by volume of leucite as a reinforcing phase. In an improvement on the Empress\textsuperscript{®} product, Ivoclar introduced the Empress 2\textsuperscript{®} ceramic restorative system. This second generation of pressable dental ceramics was the first to contain lithium disilicate as the major crystalline phase (70\% by volume), and has inspired many others including the OPC 3G\textsuperscript{®} (Pentron LLC, Wallingford, CT) pressable lithium disilicate dental ceramic. This material represented a significant increase in fracture toughness and flexural strength compared to the leucite-reinforced original Empress\textsuperscript{®}. In addition, the lithium disilicate ceramic can be heat-pressed at lower temperature (920°C), using the same laboratory equipment necessary for the pressing of the leucite-reinforced ceramic ingots. However, the lithium disilicate core does not possess the desired esthetic qualities needed to match natural teeth. The challenge then was to develop veneering porcelains that bond well to the core ceramic. The goal was to provide additional strength to the restoration while maintaining the improved esthetic properties. The original veneering ceramic developed for the Empress 2\textsuperscript{®} system contained fluorapatite as the major crystalline phase and had a firing temperature of 800°C\textsuperscript{8}. The mismatch of the coefficient of thermal expansion of the veneer ceramic to the core ceramic for Empress 2\textsuperscript{®} is -0.9x 10\textsuperscript{-6}/°C\textsuperscript{8}. The mismatch suggested in a metal-ceramic system is only -0.5x 10\textsuperscript{-6}
which may explain why the Empress 2® system suffered clinically to delamination and cracking.

The importance of the quality of the bond between the substructure and the veneering porcelain is unquestioned. Therefore, a great deal of research has been conducted to assess the quality of the metal-ceramic interface in dental restorations. The quality of the substructure-veneer bond is affected by many parameters. These include the coefficient of thermal expansion mismatch between metal and porcelain, and the cooling rate after firing. Utilizing the ductile properties of the metal, and brittleness of the porcelain, Papazoglou and Brantley were able to assess the quality of the metal-ceramic bond through porcelain adherence and force to failure. In measuring the thermal expansion of porcelain and metal, Fairhurst pointed out that values used for comparison should be collected at several temperature points up to the glass transition temperature. Guinn, et al, examined the effect of cooling rate on the apparent bond strength of a metal-ceramic couple. They demonstrated that a controlled cooling rate in a closed environment yielded higher apparent bond strengths as compared to rapid bench top cooling or very fast cooling via quenching in chilled water. Baharav, et al, measured the fracture toughness of the porcelain as a function of three cooling rates and established that a faster cooling rate yielded greater fracture toughness values. The fast cooling rate in the Baharav study was most similar to the controlled cooling rate in Guinn’s study. In a metal-ceramic system, the porcelain is more likely to exhibit fracture under these conditions due to its relative brittle nature compared to the metal.

Research into furthering all-ceramic technology has focused on extending traditional tests to ceramic systems. When these tests have been applied to homogenous
ceramic materials, the results have been variable. Ban and Anusavice studied several test methods and their influence on the failure of brittle dental materials. It was found that different biaxial test methods could yield different flexural strength values. As an alternative to these types of tests, Fischer and Marx examined and compared an indentation method with a bending test as a measure of fracture toughness for a single ceramic system. They concluded that the indentation technique may be a useful estimate of the fracture toughness for a single ceramic material, but they did not extend this idea to the assessment of the interfacial bond between ceramics of a bilayer system.

The nature of the bond strength between a core ceramic and its veneer has proven difficult to measure through techniques that have been commonly utilized in metal-ceramic testing. Similar to the metal-ceramic restoration, the bond between a veneering ceramic and a core ceramic is crucial. Without a reliable method to determine the strength of this bond, the development of compatible materials has progressed through trial and error methodology. As mentioned earlier, anecdotal reports of clinical failures of all-ceramic crowns often involve delamination of the veneering ceramic from the core. Recently, Lawn and co-workers proposed the use of a Hertzian indentation technique to analyze crack formation and damage modes in all-ceramic bilayered structures. While providing for ease of testing, the method does not provide a quantitative analysis of the interface which would permit the determination of compatible materials. Studies focusing on the quantitative determination of the bond strength between two ceramic materials combined as a porcelain veneer on a core have not been conducted. Thompson surveyed several biaxial bending methods to assess the failure mode in bilayered dental ceramics. Kelly outlined a study to evaluate the quality of the
interface through thermal shock testing\textsuperscript{23}. However, both of these studies were qualitative in scope.

Many factors can affect the interfacial bond between two dental ceramics. Upon cooling of veneered all-ceramic restorations, the veneer can be placed into tension or compression, based on the coefficient of thermal expansion difference between the core and the veneer. The heating and cooling rates used during the determination of the coefficient of thermal expansion, by dilatometry are usually low. The ASTM standard (C 372-94) recommends 3\textdegree C/minute\textsuperscript{24}. However, the cooling rates reached in a dental porcelain furnace are much higher. Therefore, determining the compatibility between veneer and core ceramics for commercial use is difficult using available test methods. Indentation techniques have long been proposed to qualitatively evaluate coating adhesion and interface toughness between brittle thin films and substrates\textsuperscript{25}. More recently, an interfacial indentation technique to measure the apparent interfacial toughness of layered materials has been described\textsuperscript{26}.

The purpose of the present study was to assess the effectiveness of a Vickers indentation technique in measuring the apparent interfacial toughness of core-veneer dental ceramic bilayers. The effect of cooling rate and thermal expansion mismatch on the apparent interfacial toughness was investigated. The hypothesis was that this information could lead to better design of all-ceramic systems and therefore better clinical longevity of these restorative systems. The null hypothesis for this study was that the indentation technique will fail to be an effective method to demonstrate differences in the apparent interfacial toughness as a result of varying rates of cooling and coefficient of thermal expansion mismatch of the core and veneering ceramics.
CHAPTER 2

MATERIALS AND METHODS

2.1 Specimen Preparation

A lithium disilicate-based pressable ceramic was used as the core material (OPC 3G®, Pentron® LLC, Wallingford, CT). Rectangular baseplate wax patterns (3x5x20 mm) were initially fabricated, sprued and invested using 100 g Universal Refractory Investment Powder, 20 mL of Universal Refractory Investment Liquid (Pentron® LLC, Wallingford, CT) and 7 mL of distilled water. The investment was hand-spatulated for 15 seconds, vacuum mixed for 60 seconds, and poured into paper rings containing the sprued wax pattern. The rings were allowed to bench set for 15 minutes. The paper and ring formers were removed and the base of the investment was leveled using a razor blade. The invested core patterns along with disposable plungers were placed in a conventional burnout furnace (Thermolyne 48000, Barnstead-Thermolyne, Dubuque, IA) preheated to 900°C, and allowed to burn out for 45 minutes.

The pressable ceramic ingots were placed in the investment rings followed by the disposable plungers. The entire assembly was then transferred to an automatic pressing machine (Optimal AutoPress, Pentron® LLC, Wallingford, CT) when the temperature
in the pressing furnace reached a level of 700°C. The furnace was raised to a temperature of 895°C, at a rate of 60°C per minute. This temperature was held for 10 minutes before pressing began. Pressing was conducted for 10 minutes under vacuum. After pressing, the rings were allowed to remain on the furnace platform until they cooled to 300°C. They were then removed and allowed to bench-cool to room temperature. The core ceramic bars were divested and sandblasted with 50 micrometer glass beads at a pressure of 2 bars to remove the investment material.

The core specimens were randomly divided into 3 groups; High, Low and Matched (n=9 per group). The core surface to be veneered was ground and sequentially polished to a 0.5 micrometer finish using silicon carbide plain back discs under water and ending with cloth discs and diamond polishing paste (LECO Corporation, St. Joseph, MI) on a polishing wheel (Rotopol-15, Struers, Rødovre, Denmark). The High group was veneered with an experimental ceramic, fabricated and provided by Pentron® LLC, of higher coefficient of thermal expansion than that of the core. The Low group was veneered with an experimental ceramic, fabricated and provided by Pentron® LLC, of lower coefficient of thermal expansion than that of the core. The Matched group was veneered with an experimental ceramic, fabricated and provided by Pentron® LLC, with a coefficient of thermal expansion matched to that of the core. The veneering ceramic was manually condensed onto the core specimens and fired using a dental porcelain furnace (Jelfire VPF, Jelenko, Armonk, NY) under vacuum from 482-773°C. The heating rate was 42°C/min with a dwell of 2 minutes at peak temperature and a total cooling time of 6 minutes on the furnace tray. The specimens were then bench-cooled to room temperature.
The High, Low and Matched groups were further randomly divided into 3 subgroups, depending on the cooling rates that the specimens were subject to. The Fast group was not exposed to further heat treatments after the veneering ceramic was fired in the dental porcelain furnace (Jelfire VPF, Jelenko, Armonk, NY). The cooling curve for the Fast group is displayed in Figure 1. The fast cooling curve was determined using a digital thermometer (Digi-Sense® Thermometer Type K Thermocouple, Cole-Parmer Instrument Company, Vernon Hills, IL) with readings taken every 20 seconds after the furnace tray descended from the furnace.

The Medium group was placed in a conventional burnout furnace and heated to 750°C at a rate of 3°C/minute and allowed to dwell for 10 minutes. The furnace was turned off and allowed to cool with the door closed to room temperature. The medium cooling curve was determined using a digital thermometer (Digi-Sense® Thermometer Type K Thermocouple, Cole-Parmer Instrument Company, Vernon Hills, IL) with readings taken every 20 minutes after the furnace was switched off (Figure 1).

The Slow group was placed in a furnace, heated to 750°C at a rate of 3°C/min and allowed to dwell for 10 minutes at peak temperature. A cooling of rate of 1°C/min was maintained until a temperature of 30°C was reached (Figure 1).
Figure 1: Fast, Medium and Slow Cooling Curves

All of the bilayered specimens were sectioned with a low speed saw and diamond blade (ISOMET, Buehler, Lake Bluff, IL) and polished sequentially to a 0.5 micrometer finish as described previously. The core ceramic was selectively etched to increase contrast from the veneering porcelain with a 0.5% hydrofluoric acid solution for 90 seconds. The interface was then indented with a Vickers hardness tester to determine the apparent interfacial toughness. Details of this experiment are given in Section 2.6.
2.2 Coefficient of Thermal Expansion

The coefficient of thermal expansion and the glass transition temperature of each group was measured by dilatometry using a single pushrod horizontal dilatometer (Orton Automatic Recording Dilatometer, Model 1600D, The Edward Orton Jr. Ceramic Foundation, Westerville, OH), in accordance with ASTM Standard C372-94\textsuperscript{24}. Bars (n=3 per group) were heated at a rate of 3°C/min. to 550°C. On cooling, the rate was set at 1°C/min to 50°C. The average differential coefficient of thermal expansion between 400 and 50°C was calculated from the cooling curve using the following equation:

\[ DCE = \frac{E_2 - E_1}{(T_2 - T_1)100} \]  

Where, \( E_1 \) and \( E_2 \) are the percent linear changes at the temperatures \( T_1 \) and \( T_2 \), respectively, and \( T_2 - T_1 = 20^\circ \text{C} \).

2.3 Hardness Testing

Vickers hardness was determined in compliance with ASTM Standard C1327-99\textsuperscript{27}, using a 136 degree diamond Vickers indenter on a hardness tester (Zwick Model 3212, Zwick GmbH & Co., Germany). The Vickers hardness number (\( H_V \)) was determined from the half diagonal of the indentations (n=10) at a load of 4.9N for the veneer ceramic, and 14.7N for the core ceramic, using the following formula:  

\[ H_V = \frac{1.854(F/d^2)}{} \]  

Where \( F \) is the applied load and \( d \) is the diagonal of the indentation.

2.4 Density

The mean density of each group (n=5) was determined via Archimedes' method using a precision scale (Analytical scale AG204, Mettler-Toledo, Inc., Highstown, NJ).
The equation for the density calculation was: \( \rho = \frac{m}{v} \), where \( \rho \) is the density, \( m \) is the mass in grams, and \( v \) is the volume of the specimen in cubic centimeters.

2.5 Modulus of Elasticity

The mean modulus of elasticity of each group (n=5) was measured using the pulsed ultrasonic velocity method (TDS3012 oscilloscope, Tektronix Inc., Beaverton, OR and Pulser-receiver Model 5072PR, Panametrics, Waltham, MA). The method involves the transit-time measurement of short wave pulses traveling over a known path through the bulk of the specimen. The ratio of the path length to the transit time yields the velocity. A frequency of 10MHz was used to measure the sound velocity in longitudinal mode (\( V_L \)) and in shear mode (\( V_S \)). From these two wave speeds and the density of the material, the elastic parameters can be calculated using the following equations:

- **Young's modulus:** \( E = \frac{\rho V_L^2 (3V_L^2 - 4V_S^2)}{(V_L^2 - V_S^2)} \)
- **Poisson's ratio:** \( \nu = \frac{(V_L^2 - 2V_S^2)}{2(V_L^2 - V_S^2)} \)
- **Bulk modulus:** \( K = \rho V_L^2 - \frac{4}{3} \rho V_S^2 \)
- **Shear modulus:** \( G = \rho V_S^2 \)

2.6 Apparent Interfacial Toughness

Interfacial indentations were performed using a Vickers indenter (Zwick Model 3212, Zwick GmbH & Co., Germany) with one diagonal of the indent oriented along the interface of the bilayered specimens (Figure 2). When using the indentation technique, it has been demonstrated that hardness increases as the applied load decreases.
Therefore, it was important to establish an optimal load for interfacial indentations. In consideration of this effect, several loads were evaluated for each group to determine the load at which interface cracking occurred most predictably. Each group was indented along the interface at loads ranging from 0.981 N to 7.848 N. The load at which interface crack propagation was most evident and predictable was determined. Indents were then performed at the critical load for each group until a total of 30 interface cracks were collected. Digital micrographs were taken immediately after indentation, using an optical
microscope (Axiotech 100HD-3D, Zeiss, Germany) equipped with a digital camera (Digital Camera DMC-1, Polaroid Corporation, Cambridge, MA). The length of the interfacial crack was measured on the digital micrographs using the public domain NIH Image software program (developed at the U.S. National Institutes of Health and available on the Internet at http://rsb.info.nih.gov/nih-image/). The apparent interfacial toughness was calculated using the following equations:

\[
K_{ca} = 0.015 \frac{P_c}{a_c} \left( \frac{E}{H} \right)^{1/2}_I \quad \text{where:} \quad \left( \frac{E}{H} \right)^{1/2}_I = \left( \frac{E}{H} \right)^{1/2}_S + \left( \frac{E}{H} \right)^{1/2}_R \\
1 + \left( \frac{H_S}{H_R} \right)^{1/2} 1 + \left( \frac{H_R}{H_S} \right)^{1/2}
\]

\[K_{ca} = \text{apparent interfacial toughness} \quad H = \text{Vickers hardness (GPa)}\]

\[P_c = \text{critical load (N)} \quad S = \text{substrate}\]

\[a_c = \text{crack length (m)} \quad R = \text{coating}\]

\[E = \text{modulus of elasticity (GPa)} \quad I = \text{interface}\]
2.7 Residual Stresses

For each Fast-cooled specimen, the residual thermal stresses were determined using the equation:\[\sigma_v = E(T_0 - T')(a_v - a_c)(1 - 3j + 6j^2)\] for the residual stress of the veneer, and
\[\sigma_c = E(T_0 - T')(a_c - a_v)j(1 - 3j + 6j^2)\] for the residual stress of the core, where:

- \(\sigma_v\) = residual stress of the veneer (MPa)
- \(\sigma_c\) = residual stress of the core (MPa)
- \(T_0\) = initial temperature (°C)
- \(T'\) = final temperature (°C)
- \(E\) = modulus of elasticity (GPa)
- \(a_c\) = CTE core
- \(a_v\) = CTE veneer
- \(v\) = veneer
- \(c\) = core
- \(j\) = thickness ratio of veneer to core

The thickness of each veneer and each core was measured using a traveling microscope (Measurescope MM-11, Nikon, Japan).

2.8 Statistical Analysis

Means for each group were analyzed by ANOVA and Tukey’s test. P-values of less than 0.05 were considered statistically significant. Parameters within the model (cooling rate and expansion mismatch) were subjected to a standard least squares regression analysis, to evaluate their weight on the apparent interfacial toughness.
CHAPTER 3

RESULTS

3.1 Coefficient of Thermal Expansion (CTE)

The CTE measurements are presented in Table 1. As expected, the CTE of the High expansion group was significantly higher than that of the other groups. The mean CTE of the core and the Matched veneering porcelain were not significantly different (p=0.196). The CTE of the Low expansion group was significantly lower than that of the other groups. The mean glass transition temperature ($T_g$) for each material was determined from the heating curve. The glass transition temperature of the core material was about 20°C higher than that of the veneering ceramic independently of the CTE. A graphic comparison of the percent linear change for the veneer ceramic to the core ceramic is shown in Figure 3 for the High CTE veneer, Figure 4 for the Matched CTE veneer, and Figure 5 for the Low CTE veneer.
<table>
<thead>
<tr>
<th>Ceramic</th>
<th>CTE ($\alpha \times 10^{-6}^\circ\text{C}$)</th>
<th>$\alpha_s - \alpha_c$</th>
<th>$T_g$ ($^\circ\text{C}$)</th>
<th>Hardness (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Core</td>
<td>9.82 (0.10)a</td>
<td>-</td>
<td>522.6 (6.1)</td>
<td>5.49 (0.04)ab</td>
</tr>
<tr>
<td>Matched</td>
<td>9.61 (0.21)a</td>
<td>-0.21</td>
<td>501.0 (1.0)</td>
<td>5.55 (0.12)ab</td>
</tr>
<tr>
<td>High</td>
<td>10.92 (0.05)b</td>
<td>1.1</td>
<td>506.7 (1.2)</td>
<td>5.41 (0.11)a</td>
</tr>
<tr>
<td>Low</td>
<td>9.11 (0.08)c</td>
<td>-0.71</td>
<td>505.3 (1.3)</td>
<td>5.60 (0.07)b</td>
</tr>
</tbody>
</table>

Identical letters indicate no significant difference at $p < 0.05$.

Table 1: Mean Values for Coefficient of Thermal Expansion, CTE mismatch, Glass Transition Temperature and Hardness ($\pm$SD).
Figure 3: Percent Linear Change for the High Veneer Group Compared to the Core Ceramic.
Figure 4: Percent Linear Change for the Matched Veneer Group Compared to the Core Ceramic.
Figure 5: Percent Linear Change for the Low Veneer Group Compared to the Core Ceramic.

3.2 Vickers Hardness

The mean Vickers hardness values for the various groups are summarized in Table 1. The mean Vickers hardness of the Low CTE veneering ceramic group was significantly higher than that of the High CTE veneering ceramic group (p=0.022) which was not significantly different from that of the core or matched veneering ceramic.
3.3 Density

The density of the Matched veneer group was significantly higher than either the Low or the Core group \((p \leq 0.001)\), but the Matched group was not significantly different from the High group \((p=0.279)\). The density of the High group was significantly higher than either the Low or the Core group \((p \leq 0.0026)\). However, the Low and Core groups showed the lowest density and were not significantly different from one another \((p=0.514)\). The mean density for each group is shown in Table 2.

3.4 Elastic Constants

The mean modulus of elasticity of the core material was significantly higher than that of all other groups \((p<0.0001)\). No significant difference was found between the Low and High veneer values \((p=0.275)\). Significant differences were also found between the Matched veneer ceramic and the High veneer \((p<0.0001)\) and Low veneer \((p=0.008)\) values. The Poisson’s ratio for the Core material was significantly higher than any of the other materials \((p<0.007)\). The Poisson’s ratio for the Low group was found to be significantly lower than any of the other materials \((p<0.019)\). The Poisson’s ratio for the Matched and the High group were not significantly different from one another \((p=0.999)\). These results are presented in Table 2.
<table>
<thead>
<tr>
<th>Ceramic</th>
<th>Density (g/cm³)</th>
<th>Young’s modulus (GPa)</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Core</td>
<td>2.463 (0.011)a</td>
<td>99.71 (0.19)a</td>
<td>0.231 (0.01)a</td>
</tr>
<tr>
<td>Matched</td>
<td>2.489 (0.006)b</td>
<td>74.67 (0.94)b</td>
<td>0.220 (0.004)b</td>
</tr>
<tr>
<td>High</td>
<td>2.479 (0.009)b</td>
<td>71.91 (0.30)c</td>
<td>0.221 (0.002)b</td>
</tr>
<tr>
<td>Low</td>
<td>2.456 (0.011)a</td>
<td>72.82 (1.17)c</td>
<td>0.211 (0.004)c</td>
</tr>
</tbody>
</table>

Identical letters indicate no significant difference at p < 0.05.

Table 2: Mean Values for Density, Young’s modulus and Poisson’s ratio (±SD).

3.5 Critical Load Determinations (Pc)

The critical load for each group was determined as the load at which the greatest amount of interfacial cracking occurred. Plots of the percentage of interfacial cracking as a function of load are displayed in Figures 6, 10, and 14 for the Fast, Medium and Slow cooling groups, respectively. The critical load was used in the subsequent interfacial Vickers indentations to determine the apparent interfacial toughness. Characteristic interfacial indentation photomicrographs for each of the cooling groups are shown in Figures 7-9, 11-13, and 15-17. It should be noted that the veneer layer is oriented above the interface in all photomicrographs, and that the scale bar in the lower right is 10 micrometers. The crack-like lines apparent in Figures 8, 9, 13, and 16 are the result of the etching process revealing the grain boundaries within the veneer layer. At the critical load, cracking was more prevalent along the interface. Cracking was more evident in the core or veneer for loads less than or greater than the critical load. The determined critical
load values are summarized in Table 3 for the Fast cooling group, Table 4 for the Medium cooling group and Table 5 for the Slow cooling group.

Figure 6: Critical Load Determination of Fast Cooling Groups (Arrows Denote $P_c$).
<table>
<thead>
<tr>
<th>Ceramics</th>
<th>High</th>
<th>Low</th>
<th>Matched</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P_c$ (N)</td>
<td>1.96</td>
<td>2.94</td>
<td>1.96</td>
</tr>
<tr>
<td>$a_c$ (μm)</td>
<td>20.14±2.95</td>
<td>22.99±1.69</td>
<td>16.91±1.51</td>
</tr>
<tr>
<td>$H_V$ (GPa)</td>
<td>5.41</td>
<td>5.60</td>
<td>5.55</td>
</tr>
<tr>
<td>$H_C$ (GPa)</td>
<td>5.49</td>
<td>5.49</td>
<td>5.49</td>
</tr>
<tr>
<td>$E_V$ (GPa)</td>
<td>71.91</td>
<td>72.82</td>
<td>74.67</td>
</tr>
<tr>
<td>$E_C$ (GPa)</td>
<td>99.71</td>
<td>99.71</td>
<td>99.71</td>
</tr>
<tr>
<td>$(E/H)^{0.5}$</td>
<td>3.95</td>
<td>3.94</td>
<td>3.97</td>
</tr>
<tr>
<td>$K_{ca}$ (MPa•m^{0.5})</td>
<td>1.34±0.29</td>
<td>1.59±0.18</td>
<td>1.70±0.25</td>
</tr>
</tbody>
</table>

Table 3: Apparent Interfacial Toughness Calculation, Fast Cooling Group.
Figure 7: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Fast Cooling, High CTE Group (1000x magnification).
Figure 8: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Fast Cooling, Low CTE Group (1000x magnification).
Figure 9: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Fast Cooling, Matched CTE Group (1000x magnification).
Figure 10: Critical Load Determination of Medium Cooling Groups (Arrows Denote $P_c$).

<table>
<thead>
<tr>
<th>Ceramics</th>
<th>High</th>
<th>Low</th>
<th>Matched</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P_c$ (N)</td>
<td>2.94</td>
<td>3.92</td>
<td>3.92</td>
</tr>
<tr>
<td>$a_c(\mu m)$</td>
<td>27.80±2.58</td>
<td>31.40±1.65</td>
<td>29.34±2.72</td>
</tr>
<tr>
<td>$H_v$ (GPa)</td>
<td>5.41</td>
<td>5.60</td>
<td>5.55</td>
</tr>
<tr>
<td>$H_c$ (GPa)</td>
<td>5.49</td>
<td>5.49</td>
<td>5.49</td>
</tr>
<tr>
<td>$E_v$ (GPa)</td>
<td>71.91</td>
<td>72.82</td>
<td>74.67</td>
</tr>
<tr>
<td>$E_c$ (GPa)</td>
<td>99.71</td>
<td>99.71</td>
<td>99.71</td>
</tr>
<tr>
<td>$(E/H)^{0.5}$</td>
<td>3.95</td>
<td>3.94</td>
<td>3.96</td>
</tr>
<tr>
<td>$K_{ca}$ (MPa*m$^{0.5}$)</td>
<td>1.21±0.17</td>
<td>1.32±0.11</td>
<td>1.49±0.22</td>
</tr>
</tbody>
</table>

Table 4: Apparent Interfacial Toughness Calculation, Medium Cooling Group.
Figure 11: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Medium Cooling, High CTE Group (1000x magnification).
Figure 12: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Medium Cooling, Low CTE Group (1000x magnification).
Figure 13: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Medium Cooling, Matched CTE Group (1000x magnification).
Figure 14: Critical Load Determination of Slow Cooling Groups (Arrows Denote $P_c$).
<table>
<thead>
<tr>
<th>Ceramics</th>
<th>High</th>
<th>Low</th>
<th>Matched</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P_c$ (N)</td>
<td>2.94</td>
<td>3.92</td>
<td>3.92</td>
</tr>
<tr>
<td>$a_c$ (μm)</td>
<td>27.31±2.42</td>
<td>31.37±0.93</td>
<td>30.96±2.22</td>
</tr>
<tr>
<td>$H_v$ (GPa)</td>
<td>5.41</td>
<td>5.60</td>
<td>5.55</td>
</tr>
<tr>
<td>$H_c$ (GPa)</td>
<td>5.49</td>
<td>5.49</td>
<td>5.49</td>
</tr>
<tr>
<td>$E_v$ (GPa)</td>
<td>71.91</td>
<td>72.82</td>
<td>74.67</td>
</tr>
<tr>
<td>$E_c$ (GPa)</td>
<td>99.71</td>
<td>99.71</td>
<td>99.71</td>
</tr>
<tr>
<td>$(E/H)^{0.5}$</td>
<td>3.95</td>
<td>3.94</td>
<td>3.97</td>
</tr>
<tr>
<td>$K_{ca}$ (MPa*m$^{0.5}$)</td>
<td>1.24±0.16</td>
<td>1.32±0.06</td>
<td>1.37±0.17</td>
</tr>
</tbody>
</table>

Table 5: Apparent Interfacial Toughness Calculation, Slow Cooling Group.
Figure 15: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Slow Cooling, High CTE Group (1000x magnification).
Figure 16: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Slow Cooling, Low CTE Group (1000x magnification).
Figure 17: Interfacial Vickers Indentations Below (a), At (b) and Above (c) the Critical Load for the Slow Cooling, Matched CTE Group (1000x magnification).

a; $P = 2.94$ N; 30% cracking along the interface.

b; $P_c = 3.92$ N; 55% cracking along the interface.

c; $P = 4.91$ N; 35% cracking along the interface.
3.6 Apparent Interfacial Toughness

The apparent interfacial toughness values were calculated using the formula proposed by Chicot, et al\textsuperscript{26}. The physical properties that were previously determined and used in the calculations for each of the cooling groups are listed in Table 3, 4 and 5. The mean values of the apparent interfacial toughness determinations are summarized in Table 6. The mean apparent interfacial toughness of the Fast cooled-Matched CTE was significantly higher than that of the other groups (p \leq 0.001), except for the Fast cooled-Low CTE group (p=0.355). The remaining groups which displayed significant differences did so based on the influence of either the cooling rate, or the coefficient of thermal expansion mismatch, or both. These factors are considered in Chapter 3.8.

<table>
<thead>
<tr>
<th>Groups</th>
<th>High</th>
<th>Low</th>
<th>Matched</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fast (MPa·m\textsuperscript{0.5})</td>
<td>1.34±0.29abcde</td>
<td>1.59±0.18gh</td>
<td>1.70±0.25h</td>
</tr>
<tr>
<td>Medium (MPa·m\textsuperscript{0.5})</td>
<td>1.21±0.17a</td>
<td>1.32±0.11abcd</td>
<td>1.49±0.22g</td>
</tr>
<tr>
<td>Slow (MPa·m\textsuperscript{0.5})</td>
<td>1.24±0.16ab</td>
<td>1.32±0.06abc</td>
<td>1.37±0.17abcdef</td>
</tr>
</tbody>
</table>

Identical letters indicate no significant difference at p <0.05.

Table 6: Mean Apparent Interfacial Toughness Values (MPa·m\textsuperscript{0.5}).
3.7 Residual Stress Determinations

Residual stresses for each veneer and each core were determined using Kingery’s equations for glaze stresses\(^{30}\). This equation assumes that no stress relaxation occurred during cooling and therefore it is only applicable to the fast-cooled groups. Positive values for residual stresses indicate that tension is present within the material, while negative values indicate that the material is in a state of compression. The results are presented in Table 7.

<table>
<thead>
<tr>
<th>Rate</th>
<th>Groups</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fast</td>
<td>Veneer</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>High</td>
<td>290.5</td>
<td>337.0</td>
<td>211.5</td>
</tr>
<tr>
<td></td>
<td>Low</td>
<td>-165.1</td>
<td>-159.9</td>
<td>-183.1</td>
</tr>
<tr>
<td></td>
<td>Matched</td>
<td>-40.7</td>
<td>-35.0</td>
<td>-36.6</td>
</tr>
<tr>
<td>Core</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>High</td>
<td>-539.1</td>
<td>-667.9</td>
<td>-341.1</td>
</tr>
<tr>
<td></td>
<td>Low</td>
<td>284.4</td>
<td>271.6</td>
<td>330.1</td>
</tr>
<tr>
<td></td>
<td>Matched</td>
<td>63.8</td>
<td>51.4</td>
<td>54.8</td>
</tr>
</tbody>
</table>

Table 7: Calculated Residual Stresses of the Veneer and Core Ceramics for each Specimen (MPa).
3.8 Least Squares Regression Analysis

The apparent interfacial toughness was affected by both the cooling rate and the coefficient of thermal expansion mismatch. To better understand the influence of the cooling rate and the coefficient of thermal expansion mismatch between veneer and core, ANOVA and standard least-squares regression analyses were performed. The results of these analyses are presented in Tables 8 and 9. The influence of each independent variable is illustrated in Figure 18 and 19. Each data point in these figures represents the apparent interfacial toughness for each interfacial indentation measured in this study. The mean apparent interfacial toughness for all interfacial indentations is represented by the horizontal line and was 1.398 MPa·m$^{0.5}$. The relative effect of the cooling rate or the CTE mismatch on the apparent interfacial toughness can be seen by the relation of the mean diamond (sample mean and 95% confidence interval) to the horizontal mean apparent interfacial toughness line. A fast cooling rate had higher apparent interfacial toughness among all groups, and was significantly higher for Matched and Low CTE mismatch variables. A Matched CTE led to increased apparent interfacial toughness compared to the Low CTE and High CTE mismatches regardless of cooling rate. The High CTE mismatch led to significantly lowered toughness compared to the Matched CTE. From the standard least squares analysis, a prediction formula was derived that provides an indication of the effect and intensity for each variable on the apparent interfacial toughness. This formula is given in Table 10. The fast cooling rate would increase the toughness value by an estimated 0.145 MPa·m$^{0.5}$ (p<0.0001). The coefficient of thermal expansion mismatch had a significant effect on the toughness, as illustrated by the matched group, which exhibited an estimated increase in the apparent
interfacial toughness of 0.123 MPa•m$^{0.5}$. The residual value ($r^2 = 0.407$) indicates that there is a reasonably good fit between the observed apparent interfacial toughness and predicted values for the lithium disilicate ceramic studied, as shown in Figure 20.
Figure 18: Influence of Cooling Rate on Apparent Interfacial Toughness (MPa\*m^{0.5}).
Figure 19: Influence of CTE Mismatch on Apparent Interfacial Toughness (MPa·m$^{0.5}$)
<table>
<thead>
<tr>
<th>Combined</th>
</tr>
</thead>
<tbody>
<tr>
<td>R Square</td>
</tr>
<tr>
<td>Root Mean Square Error</td>
</tr>
<tr>
<td>Mean of Observations</td>
</tr>
<tr>
<td>Sum Weights</td>
</tr>
</tbody>
</table>

Table 8: Summary of Fit for ANOVA.

<table>
<thead>
<tr>
<th>DF</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>Prob &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cooling</td>
<td>2</td>
<td>2.90</td>
<td>1.45</td>
</tr>
<tr>
<td>Expansion</td>
<td>2</td>
<td>3.06</td>
<td>1.53</td>
</tr>
<tr>
<td>Cooling*Expansion</td>
<td>4</td>
<td>0.53</td>
<td>1.33</td>
</tr>
<tr>
<td>Error</td>
<td>261</td>
<td>9.45</td>
<td>0.05</td>
</tr>
<tr>
<td>C Total</td>
<td>269</td>
<td>15.96</td>
<td>0.06</td>
</tr>
</tbody>
</table>

Table 9: Analysis of Variance.

<table>
<thead>
<tr>
<th>Ksc</th>
<th>Cooling Rate</th>
<th>Expansion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.40</td>
<td>0.15 (Fast)</td>
<td>-0.14 (High)</td>
</tr>
<tr>
<td>+</td>
<td>-0.06 (Medium)</td>
<td>+</td>
</tr>
<tr>
<td>-0.09 (Slow)</td>
<td>+</td>
<td>0.12 (Matched)</td>
</tr>
</tbody>
</table>

Table 10: Prediction Formula for Apparent Interfacial Toughness by Variable.
Figure 20: Predicted versus Observed Apparent Interfacial Toughness.
CHAPTER 4

DISCUSSION

The quality of the adhesion between two ceramic materials joined in a bilayer is influenced by both intrinsic and extrinsic factors. Intrinsic factors include the physical properties of both ceramics such as the elastic constants, the toughness of both materials, the coefficient of thermal expansion and the glass transition temperature. Extrinsic factors that can have an influence on the all-ceramic system include the cooling rate after veneer sintering and the core to veneer thickness ratio. The purpose of this study was to evaluate the effect of cooling rate and coefficient of thermal expansion mismatch on the apparent interfacial toughness of an all-ceramic core-veneer system.

The results showed that the two groups with the highest mean apparent interfacial toughness values were the Fast cooling - Matched CTE (1.70±0.25 MPa•m$^{0.5}$) group, followed by the Fast cooling - Low CTE mismatch (1.59±0.18 MPa•m$^{0.5}$) group. While these two groups were not statistically different from one another, the Fast cooling - Matched CTE group was significantly higher than all of the remaining groups. In terms of the apparent interfacial toughness, the third and fourth highest groups were the Medium cooling – Matched CTE and the Slow cooling – Matched CTE groups. They were not found to be different from one another. The five lowest groups were found to
have mean apparent interfacial toughness values statistically similar to each other, but significantly lower than the four groups described above. ANOVA and standard least squares regression analyses indicated that the two factors that had the greatest influence on the apparent interfacial toughness were whether the cooling rate was fast or slow (p<0.001), and whether the CTE of the veneer was high or matched (p<0.001). The regression formula predicted the highest apparent interfacial toughness with a fast cooling rate and matched coefficients of thermal expansion. It also anticipated a decrease in apparent interfacial toughness with a medium and a slow cooling rate with either high or low expansion, although the low expansion factor had a very minimal impact.

As mentioned earlier, the apparent interfacial toughness measured in this study depended on the presence of residual thermal stresses in the ceramic system. The intensity of these stresses is directly related with the cooling rate while their quality (compressive or tensile) is dictated by their thermal expansion mismatch and thickness ratio of the two layers. Our results emphasized the role of the cooling rate on the development of residual thermal stresses in bilayered ceramics. Both the cooling rate and temperature range experienced through cooling have been demonstrated to have an effect on the formation and intensity of residual stresses\textsuperscript{31,32}.

High cooling rates lead to the formation of greater residual stresses\textsuperscript{33}. Conversely, slow cooling rates allow residual stress relaxation, leading towards a stress-free material. Asaoka, et al., confirmed these findings of increased residual stresses from increased cooling rate\textsuperscript{34}. In a study of metal-ceramic systems composed of CTE mismatched materials cooled at various rates, Coffey, et al., found that slowly cooled specimens showed significantly lower flexural strength than rapidly cooled specimens in
a four-point bending test\textsuperscript{35}. They concluded that this was due to the slow cooling rate which minimized the formation of favorable compressive residual stresses in the ceramic. They also showed that a fast cooling rate was effective in reducing the effect of undesirable CTE mismatches, as compared to the slow and medium cooling rates. Similarly, Anusavice and Gray showed that slow cooling rates resulted in more frequent development of delayed fractures and cracking\textsuperscript{36}. These findings suggest that fast cooling rates are more desirable for the formation of residual compressive stresses. These conclusions correlate well with the results of the present study, which were that a faster cooling rate developed increased compressive residual stresses, resulting in higher apparent interfacial toughness values. However, it is also well known that thermal shock can result in porcelain failure\textsuperscript{23}. Guinn, et al., showed that rapid cooling of metal-ceramic restorations from a dental furnace to an ice chest resulted in significantly lower apparent bond strength compared to a controlled cooling environment\textsuperscript{13}. The measurement of linear expansion in a dilatometer is the accepted method to determine the coefficient of thermal expansion\textsuperscript{24}. In this environment, the cooling rate is highly controlled at 1\textdegree C/min. However, in the fabrication of veneered all-ceramic restorations, the cooling rate is drastically increased, compared to that of the dilatometer, due to the use of a dental furnace. Our results also showed that relying only on thermal expansion measurements to determine the compatibility between core and veneering ceramics may not constitute the best approach due to the difference in cooling rates between dilatometer and dental porcelain furnaces. The indentation technique used in this study could provide more accurate information since the cooling rate can be made the same as in a dental porcelain furnace. Mackert and Evans demonstrated that slower cooling rates for leucite containing
porcelains resulted in increased leucite crystal formation which caused an increase in the coefficient of thermal expansion such that a mismatch was created. A similar mechanism is possible in our system, with formation or growth of lithium disilicate crystals leading to a mismatch in thermal expansion between core and veneer and decreased apparent interfacial toughness.

Due to its brittle nature, ceramic is much more resistant to fracture when it is in a state of compression. It is therefore beneficial when favorable residual compressive stresses are present in the veneering ceramic. Residual tensile stresses often result in the failure of the system at the interface, causing delamination or fracture of the veneering ceramic due to delayed crack formation. Anusavice, et al, studied the problem of delayed crack formation and failure within a metal-ceramic system. They found that delayed crack formation can occur up to several weeks after fabrication due to the incorporated residual tension.

As was expected, the results revealed that for all-ceramic systems, a veneering ceramic with a high CTE should be avoided. The intentionally mismatched specimens formed for the High group evidenced delamination at the margins of the specimens. The fast that they evidenced significantly lower apparent interface toughness values than the Fast Low, Fast Matched and Medium Matched Groups corroborated this macroscopic observation.

As mentioned earlier, an intrinsic factor that could influence apparent interfacial toughness is the glass transition temperature. Stresses can develop in a material during heating as well as during cooling. It is those that form below the glass transition temperature during cooling which are relevant in the fabrication of veneered ceramic
dental restorations. In this study the glass transition temperature was found to be 504 ±2.75°C for the fluorapatite veneer ceramics, and 522 ±6.11°C for the core ceramic. These figures compare with published values of 515 ±10°C for the veneer ceramic and 535 ±10°C for the core ceramic. Above the glass transition temperature of the veneering ceramic, the veneer material is in a relaxed state, which allows it to accommodate and disperse stresses. Anusavice and Hojjatie demonstrated that a rapid cooling rate can form residual compressive stresses which significantly increase the fracture toughness of veneering porcelains. In addition, the core material of the restoration also develops residual stresses on cooling.

As long as the coefficient of thermal expansion of the veneer is lower than that of the core ceramic, these stresses should be less important at the interface than the residual stress generated within the veneer. To an extent, this is taken into account in Kingery’s equation which factors in the relative thickness of each layer in his residual stress equation. Wakabayashi and Anusavice supported this, by finding that as the ratio of the core to the veneer increases, the greater the influence of the residual stress developed in the veneer, due to the shifting of the crack initiation site into the core. The core to veneer thickness ratio has an important effect on the distribution of the residual stress formed during cooling. Kingery incorporated the thickness of each layer into his equation for residual stresses. Thompson demonstrated that an increased core to veneer ratio would extend the influence of the residual stress from the veneer beyond the interface into the core material. Our results implied that residual compressive stresses were present in the veneer ceramic for the Low and Matched Fast-cooled groups. That could explain the higher mean apparent interfacial toughness values recorded for those
One last extrinsic factor that could affect the apparent interfacial toughness is the surface roughness of the core. Lubovich and Goodkind demonstrated that mechanical retention offered by the metal core can have significant positive effects on the quality of the bond formed with the veneer\textsuperscript{41}. In the present all-ceramic study, the surface of the core prior to veneering was polished to a 0.5 micrometer finish to eliminate mechanical retention as a mechanism for interfacial bonding and permit indentations at the interface in a more consistent manner.

Overall, the physical properties of the materials studied compared well with published values. The Young's modulus of the lithium disilicate core ceramic was found to be 99.7 GPa, which compared well with a published value of 104 GPa\textsuperscript{42}. The hardness values attained ranged between 5.41 GPa and 5.6 GPa for the veneer ceramic and 5.49 GPa for the core ceramic. These values are also consistent with published values of 5.55 GPa for the fluorapatite veneer\textsuperscript{43} and 5.3 GPa for the core materials\textsuperscript{44}. The density values between 2.45 and 2.49 g/cm\textsuperscript{3} obtained for the different veneering ceramics used in this study are comparable to published values\textsuperscript{45}. Variations in the density of the sintered glass-ceramic veneer are likely attributable to the condensation technique used during the formation of the veneer test specimens\textsuperscript{46}. For the core ceramic, the density exhibited little variation, possibly due to the commercially manufactured ingot and the heat pressing technique which minimized the potential for density changes.

Studies on the fracture toughness of all-ceramics systems continue today\textsuperscript{47}. The fracture toughness is the resistance of a material to fracture, and is a measure of the amount of energy necessary to cause fracture. Lithium disilicate core ceramics have been
shown to have a fracture toughness of $2.7 - 3.2 \text{ MPa}\cdot\text{m}^{0.5}$. In a study on failure origins of all-ceramic dental materials, Thompson observed that the flexural strength of the core material generally exceeded that of the interface toughness. In addition, the fluorapatite veneering ceramic is a sintered ceramic that has been shown to have a fracture toughness of $1.3 \text{ MPa}\cdot\text{m}^{0.5}$. It would be expected that the apparent interfacial toughness would not fall below the value of the veneering material. The results of the present study confirm this assumption.

The Vickers indentation technique has been applied to measure the fracture toughness of ceramic materials and is relatively straightforward and simple. It allows for many indents to be done on a relatively small specimen. This technique can be considered advantageous over other techniques for these reasons. However, using this technique to analyze the interface of all-ceramic dental materials has only been conducted on a limited basis. Bhattacharya and Petrovic utilized a Vickers indentation test oriented at several angles relative to the interface to assess the quality of an all-ceramic interface. In a similar effort to evaluate the mechanical properties of the interface, Gopal et al., applied a Vickers indentation adjacent to the interface and observed subsequent cracking for deflection caused by the interface. These methods only provide qualitative assessments of the interface. To gain better quantitative knowledge about the interface, several studies have been performed. Chiang, et al., used a Vickers indentation technique, applied in a plane perpendicular to the interface, to measure the adhesion for thin films. As a result of indentation, interface fracture toughness was determined from the resulting lateral cracking. Chicot, et al., proposed a technique to measure the apparent interfacial toughness of bilayered ceramics. In this technique they
aligned the Vickers indenter along the interface so that cracking would propagate at the interface. The resulting interfacial crack length could then be measured to assess the apparent interfacial toughness. Using this Vickers indentation method, our results showed that the mean apparent interfacial toughness values ranged from 1.21 – 1.70 MPa•m^{0.5} for core-veneer dental bilayers. Higher apparent interfacial toughness values were evident in fast cooling groups that contained veneer CTE mismatch values of -0.21 x10^{-5}/°C compared to the core ceramic. This supports the assumption that established CTE mismatches for metal-ceramic restorations are suitable guidelines for lithium disilicate all-ceramic dental restorations.

The results of the present study support the application of the Vickers indentation technique for measuring apparent interfacial toughness. The faster cooled groups of matched or low mismatch CTE differences showed higher apparent interfacial toughness values. The standard least squares regression analysis indicated that the fast cooling rate had the most influence on the apparent interfacial toughness. This was followed closely by the influence of the coefficient of thermal expansion. These results make sense, in that the potential for residual stresses to develop is greater in fast cooling environments, as has been previously demonstrated^{39,40}. Therefore, the Vickers indentation technique utilized here was effective in revealing differences between groups.
CHAPTER 5

CONCLUSIONS

The stated null hypothesis for this study was that the indentation technique would fail to be an effective method to demonstrate differences in the apparent interfacial toughness as a result of varying cooling rates and thermal expansion mismatch between core and veneering ceramic. This hypothesis was rejected and significant differences in apparent interfacial toughness were found as a function of cooling rate and CTE mismatch. Within the limitations of this study, the following conclusions can be drawn from the results:

1. This Vickers indentation test was an effective method for determining significant differences in apparent interfacial toughness values of bilayered dental ceramic systems.
2. A fast cooling rate led to a significantly higher apparent interfacial toughness than that obtained with the medium or slow cooling rates for both Matched and Low CTE groups.
3. The mismatch in CTE resulted in a significantly lower apparent interfacial toughness for the groups with a high CTE mismatch between the veneer and core.
4. The cooling rate had a greater effect on the apparent interfacial toughness than the mismatch in coefficient of thermal expansion.
REFERENCES


