A COMPARISON OF TENSILE BOND STRENGTH BETWEEN LOW TRANSLUCENCY AND HIGH TRANSLUCENCY LITHIUM DISILICATE CERAMICS USING TWO DIFFERENT RESIN CEMENTS

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CERTIFICATE OF APPROVAL

MASTER'S THESIS

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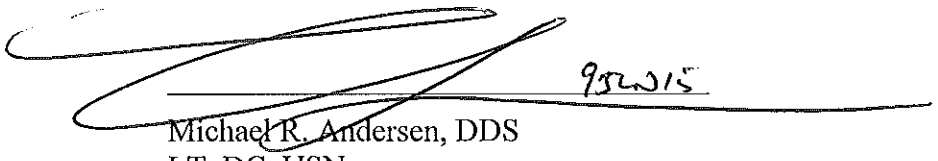

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ABSTRACT

Objectives: To compare two different dual-curing resins cement interfaces between low translucency and high translucency lithium disilicate glass-ceramics in vitro.

Materials and Methods: ISO 11405 was modeled by using a standardized dumb-bell test to compare the tensile bond strength of two different resin cement interfaces of a low translucency lithium disilicate ceramic to a high translucency lithium disilicate ceramic. The two different dual-cured resin cements were tested: Panavia F 2.0 and Variolink 2. The testing used ten dumbbell specimens for each respective group of resin cement. A total of 20 half-dumbbell shaped ceramic specimens were low translucency shade A2 lithium disilicate and 20 half-dumbbell shaped ceramic specimens were high translucency A2 lithium disilicate. Al bonding protocols were followed as instructed by the manufacturers. The respective resin cements were then applied to the treated surfaces of each half-dumbbell specimen and placed in a standardized index jig that incorporated a 0.10mm gap size between ceramics. The dumbbell specimens were placed in a MTS Insight 5kN load cell with customized titanium grips and placed under tension at a crosshead speed of 0.5mm/min until the resin interface was broken. Data collected was, peak stress, strain at break, modulus of elasticity, and peak load.

Results: The mean Peak Stress (MPa) for Panavia F2.0 was 9.77 and Variolink 2 was 12.15 ($p = 0.853$). The mean Strain at Break (%) for Panavia F2.0 was 4.06 and Variolink 2 was 1.99 ($p = 0.315$). The mean Modulus of Elasticity (GPa) for Panavia F 2.0 was 0.51 and Variolink 2 was 0.62 ($p = 0.436$). The mean Peak Load (N) for Panavia was 184.07 and Variolink 2 was 238.42 ($p = 0.739$). No statistical difference between the two groups was

found, so the null hypothesis could not be rejected.

Conclusions: The tensile bond strength of two resin interfaces was compared: Panavia F 2.0 and Variolink 2. The groups tested were CAD-CAM manufactured low translucency lithium disilicate glass-ceramic specimens resin bonded to CAD-CAM manufactured high translucency lithium disilicate specimens. Because of the promising physical and optical properties of lithium disilicate, the ceramic-resin-ceramic interface can be a promising restorative option for the clinician and the patient. Clinical examples of this interface are the ceramic customized abutment resin bonded to a ceramic crown as well as a ceramic veneer overlay resin-bonded to a ceramic core substrate. Although there appears to be a gap in the literature that explores this interface, this in vitro study does not reject the difference in tensile bond strength between two commonly use dual-cured resin cements.

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LIST OF ABBREVIATIONS

CAD	.....	Computer-Assisted Design
CAM	.....	Computer-Assisted Manufacturing
HT	.....	High Translucency
LT	.....	Low Translucency
TBS	.....	Tensile Bond Strength

INTRODUCTION

Lithium disilicate is a recently developed glass-ceramic indicated for the fabrication of dental restorations by Computer-Aided Design / Computer-Aided Manufacturing (CAD/CAM) milling or a lost wax, heat pressed method. Veneering a dental ceramic restoration involves the removal of a portion of the bulk ceramic material and subsequent application of manually applied porcelain. Whether CAD/CAM manufactured or heat pressed, lithium disilicate restorations can be veneered with a fluorapatite based porcelain in a stackable powder-liquid form. Manually layering powdered porcelain, also referred to as stacking, to an all ceramic restoration is labor intensive and requires a highly skilled laboratory technician who must meticulously comply with the manufacturer's specific instructions for a quality restoration. Despite the increased optical result of veneering an all ceramic restoration, there is a consequent change of the physical material properties of the restoration and potential for increased risk of chipping, fracture, or even delamination of the layered ceramic. To date, there is a lack of evidence specifically evaluating the long-term clinical outcomes of veneered lithium disilicate restorations compared to the monolithic restoration. There is also a gap in the literature investigating the potential benefits of veneering a lithium disilicate restoration with a resin bonded lithium disilicate overlay veneer (Figure 1).

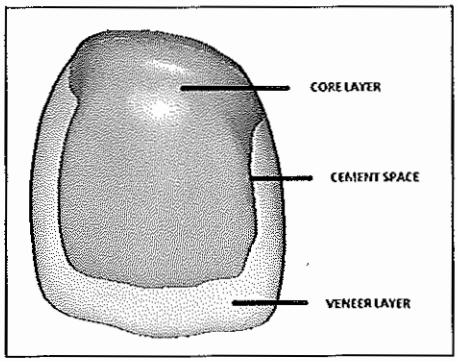

Figure 1. Computer animated design of a veneered dental restoration.

Lithium disilicate has also been shown to demonstrate a predictable, long term bond to resin cement. Since lithium disilicate restorations are available in various shades and translucencies, there may be potential esthetic or material property benefits of resin bonding a lithium disilicate substrate to a second lithium disilicate veneer overlay. Additionally, CAD/CAM manufactured lithium disilicate substrates and a CAD/CAM overlay veneer may have potential benefits compared to heat pressed lithium disilicate substrates and overlays. CAD/CAM manufactured lithium disilicate restorations are mostly automated and has great potential for a consistent product, whereas heat pressing lithium disilicate glass-ceramics involves multiple, labor intensive laboratory procedures prone to human error if manufacturer's instructions are not methodically followed.

Commercially, lithium disilicate glass-ceramics are increasingly popular due to their physical and optical properties and have recently expanded their functional use as customized implant abutments and even full contoured dental implant restorations. In the former example, customized implant abutments are tightened onto the platform of the implant with a small internal screw, and then an all-ceramic crown is resin delivered with

resin cement (Figure 2). However, lithium disilicate has only been recently available for fabricating these types of customized dental implant abutments, and therefore there is a lack of long term clinical evidence to support their use.

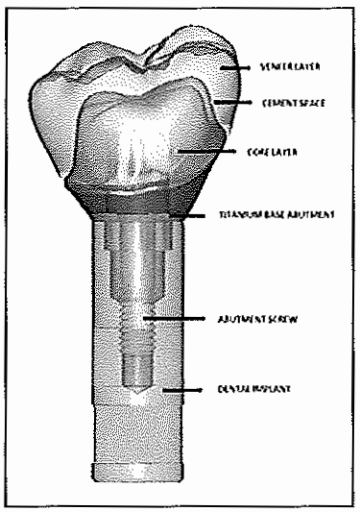

Figure 2. Computer animated design of a cementable dental implant restoration using a titanium base and customized abutment.

Since resin cement and glass-ceramics are weakest in tension by nature, tensile bond strength is a standard measurement in assessing long term bond strength by testing the resin-ceramic interface. Due to the increased and expansive use of lithium disilicate glass-ceramics in addition to the potential benefits and apparent gap in the literature involving the resin bond of two CAD manufactured lithium disilicate interfaces, this study will therefore investigate the tensile bond strength (TBS) of a low translucency (LT) CAD lithium disilicate substrate resin bonded to a high translucency (HT) CAD lithium disilicate overlay veneer.

REVIEW OF THE LITERATURE

According to Craig (2006), the term 'ceramic' refers to "any product made from a nonmetallic inorganic material usually processed by firing at a high temperature to achieve desirable properties". Dental porcelains are specialized ceramics used by dental technicians to fabricate lifelike crowns, bridges and veneers. Ceramics have a crystalline structure composed of nonmetallic inorganic materials such as metal oxides, carbides, nitrides, borides, and a complex mixture of these materials (Kingery, 1976). Although ceramics are inherently very strong, they are also inherently brittle and can fail catastrophically under tensile forces. Therefore, dental porcelains can be reinforced with other particles such as leucite or lithium disilicate crystals (Beham, 1990) to increase flexural strength. These reinforcements form interlocking microstructures that strengthen the porcelain and limit crack propagation (Apel & colleauges, 2008). Research on lithium-disilicate was first presented to the American Ceramic Society in San Francisco, California on October 31, 1973 (Borom & Turkalo, 1975). Their work examined Li_2O-Al_2O_3-SiO_2 glass ceramic to characterize the microstructures produced by variations in heat treatment and interpret their physical properties. They discovered that depending on the heat-treatment schedule, lithium disilicate (Li_2Si2O_5) and lithium metasilicate (Li_2SiO_3) were the major portions of crystalline material and that lithium disilicate had a greater effect on the stress-to-fracture of the glass composite than lithium metasilicate. Borom and Turkalo theorized that lithium disilicate crystals enhanced the strength of the parent glass due to the compressive stresses in the glass resulting from the high-expansion-coefficient crystal and lower-expansion-coefficient glass.

Lithium-disilicate ceramic dental restorations were commercially available in 1999 as Empress 2 (Ivoclar North America) and were later reformulated in 2005 as IPS e.max Press® (Ivoclar Vivadent) for increased physical and optical properties. This experimental ceramic showed no cracks with increasing wear cycles and demonstrated less wear upon opposing tooth structure than the other all-ceramic materials tested (Etman, 2009). Etman concluded that the experimental lithium disilicate showed the highest resistance to crack formation and propagation likely due to the high volume of crystalline phases in the material.

Lithium-disilicate ceramics can be used as dental restorations monolithically or veneered with a fluorapatite glass for esthetics. Due to the strength of the material, the dentist also has the option of either conventional cementation or bonding the ceramics, although studies have shown that adhesive bonding increases the fracture resistance of ceramic restorations (K. A. Malament, Socransky, Thompson, & Rekow, 2003; Sorensen, 1999; Wolf, Bindl, Schmidlin, Lüthy, & Mörmann, 2007).

FABRICATION TECHNIQUES FOR DENTAL CERAMIC RESTORATIONS

Most dental ceramics are composite structures consisting predominantly of glasses, porcelains, glass-ceramics, or highly crystalline structures (Anusavice, 2003). The variations of microstructure and fabrication methods of dental ceramics affect their physical characteristics and ultimately their application in restorative dentistry (Giordano & McLaren, 2010). Due to the multitude of ceramics available, there are many ways of fabricating ceramic dental restorations.

Powder condensation is the conventional "powder and water" stacking porcelain. Although the outcome can be very esthetic, the physical properties of this type of porcelain are traditionally weak, generally due to the large amount of residual porosities after vitrification. Historically, this method has been used for full crowns, inlays, and onlays; however, since the advent of stronger ceramics like lithium disilicate or zirconia, this fabrication method today is typically used as a monolithic veneer, veneering layer for polylithic ceramic restorations, and for veneering metal-ceramic restorations because of the esthetic optical properties (J. Kelly, 1997).

Slip casting is a technique sensitive process of fabricating porcelain restorations that involves a negative replica framework, usually gypsum, to extract the excess water from the slip through capillary action. A slip is a low-viscosity mixture of powdered ceramic in a water suspension. From this stage, the excess slurry slip is discarded and the residual ceramic within the negative gypsum framework is partially or fully sintered. Sintering occurs when the ceramic becomes a coherent mass at very high temperatures without melting (McLean, 2001). Typically in ceramics, the powdered porcelains are held at a temperature just below the melting point to allow the atoms in the powder to fuse together to create one solid piece. Sintering under vacuum has been shown to greatly increase density of the porcelain and decrease porosities. Although porcelains processed by slip casting tending to have reduced porosities, fewer processing defects and higher toughness than conventional feldspathic porcelains, slip casting has limited application in dentistry today because of the complicated and difficult steps involved in fabrication (Denry, 1996).

Hot pressing ceramics utilizes the traditional lost wax method of fabrication. This method heats a prefabricated ceramic ingot to a viscous liquid, which is then slowly pressed to form under vacuum. The microstructure of these ingots is similar to powder porcelains typically reinforced with lithium-disilicate, leucite, or fluoroapatite. Porosities within the material are significantly reduced due to the viscosity and pressure during fabrication. Hot pressed ceramics are typically much stronger than powder condensation ceramics, but also can be less esthetic. Due to the optical properties and increased strength, hot pressed ceramics are recommended for veneers, inlays, onlays, single unit crowns and short spanning anterior fixed dental prostheses (Mrazek, 1997).

CAD/CAM is a subtractive machining technique using computerized milling with a digital "impression" for fabricating ceramic dental restorations. After the tooth has been prepared, an optical impression is made intraorally or on the stone cast. This produces a digital image of the proposed restoration for the dentist or technician to digitally customize any aspect of the restoration with the software. After the appropriate modifications have been made, the milling apparatus mechanically prepares the ceramic restoration from an industrially fabricated ingot of the ceramic of choice with high speed diamond burs (Tinschert, Natt, Hassenpflug, & Spiekermann, 2004). The use of dental CAD/CAM technology has increased significantly since its inception (Poticny & Klim, 2010). In 2011, more than an estimated 30,000 dentists worldwide own a scanning and milling machine (Davidowitz & Kotick, 2011). Today, there are many choices of scanning and milling systems for CAD/CAM ceramics in the dental office or in a commercial dental laboratory.

HISTORY OF CAD/CAM AND CAD/CAM FABRICATION OF LITHIUM DISLICATE RESTORATIONS

Dr. Francois Duret invented the first CAD/CAM device for dental purposes in 1971 (Tinschert, Natt, Hassenpflug, & Spiekermann, 2004). He produced the first dental CAD/CAM restoration in 1983 and demonstrated its use at the French Dental Association's international congress in November 1985 (Davidowitz, 2011). The original Duret system (Duret CAD-CAM, Henson International) and its derivative (the Sopha system, Bioconcept, Inc.) were highly sophisticated, but were not commercially successful (Heymann & Bayne, 1996) due to the technical difficulty of intraoral imaging and critical limitations of the system (Miyazaki & colleagues, 2009).

Drs. Werner Mörmann and Marco Brandestini placed the first chairside ceramic dental inlay restoration on Sept. 19, 1985 at the University of Zurich Dental School (Mörmann, 2006). Drs. Mörmann and Brandestini are credited with the development of the first commercial CAD/CAM system, CEREC, an acronym for "Ceramic Reconstruction." Advantages of CAD/CAM restorations include repeatability, quick fabrication time, and digitization of information. However, one of the major disadvantages to the earlier CEREC systems was a large marginal fit discrepancy due to the limitations of the camera's accuracy to digitally capture the three dimensional contours of the preparations (Miyazaki & colleagues, 2009).

Although the earlier CAD/CAM technology was limited to veneers, inlays, onlays, and full crowns, dentists today can utilize CAD/CAM technology for fabricating fixed dental prostheses, milling prepolymerized resin for complete denture bases, milling

titanium or zirconia for implant abutments, creating maxillofacial prosthetics, and orthodontic appliances (Davidowitz, 2011). The advent and continual improvement of CAD/CAM technology have increased dental restorative treatment options.

ADVANTAGES OF CAD/CAM FABRICATED LITHIUM DISILICATE RESTORATIONS

CAD/CAM offers the possibility of fabricating an esthetic and functional ceramic restoration in a single patient visit (Wolf & colleauges, 2007). Due to the accuracy of the machinery and low shrinkage of the lithium disilicate glass-ceramic (0.2%), intraoral corrections are seldom necessary. According to Wiedhahn (2007), the entire treatment process lasts around two hours and leaves the milling and firing times available for other treatments. CAD/CAM restorations also allow the option for the potential added benefit of immediate bonding to freshly cut dentin and enamel (Magne, Kim, Cascione, & Donovan, 2005). These significant advantages of CAD/CAM can potentially lower cost of treatment due to fewer patient visits and less time in the dental chair. There are also fewer laboratory steps and more standardized machinery compared to the heat press method, leading to less chance for human error.

CAD-manufactured lithium disilicate is milled during the "blue block" stage, a precrystallized stage allowing the material to be milled easily without excessive damage to the material or the diamond milling burs. In this stage, the lithium metasilicate crystals are precipitated, creating the blue color. After milling, the final crystallization occurs in an 850°C vacuum oven, which dissolves the metasilicate crystal phase completely and

crystallizes the lithium disilicate, resulting in 70 percent by volume crystal ceramic with the preselected shade (D. Fasbinder & Dennison, 2010)

DISADVANTAGES OF CAD/CAM FABRICATED LITHIUM DISILICATE RESTORATIONS

The manufacturing methods of CAD/CAM ceramics have been shown to induce internal stress and cause external surface damage to the ceramic (Sindel & colleagues, 1998; Tsitrou & colleagues, 2007). The resultant flaws produced by the milling processes leaves a damage zone of 40 to 60 μm, which may be the predominant cause for the reduction in flexural strength (Sindel & colleagues, 1998), and may leave residual stresses within the ceramic. Internal stress and external surface damage significantly reduce the fracture toughness of ceramics (Lawn & colleagues, 2004). One study concluded that industrially prepared ceramics are more homogenous and structurally reliable than heat-pressed ceramics, although CAD/CAM procedures induce surface and subsurface flaws that may adversely affect the material (Tinschert, Zwez, Marx, & Anusavice, 2000). Another study demonstrated that the mean flexural strength of a CAD/CAM machined glass-ceramic can be increased by 40% with adhesive bonding, but there are remaining additional failure origins that cause the eventual fracture of the ceramic (Sindel et al., 1998).

Furthermore, the intaglio surface and marginal fit of CAD/CAM ceramics is limited by the geometry of the preparation, optical accuracy of the scanner, and the precision of the milling machine (Tinschert, Natt, Hassenpflug, & Spiekermann, 2004). The first commercial ceramic milling machines used a single diamond-grinding wheel; current machines use three-, four-, five-, six, and seven-axis CAM system diamond burs.

Subsequently, the surfaces of the ceramic restorations are able to be milled more precisely with increased axes and angles of the diamond burs. The fewer the axes, the more likely a 'stair-stepped' or 'over-milled' surface will result. The resulting surface defects have a poor adaptation to the tooth abutment and can be focal points for tensile stress within the ceramic (Tsitrou, Northeast, & van Noort, 2007).

Powder used to acquire the intraoral image impression can accumulate 20 to 56 microns and up to 600 microns in some areas of the cavity preparation, leading to error in marginal and intaglio adaptation of the restoration (E. Rekow, 1993). Regarding the internal fit of all-ceramic crowns, Lee and colleagues (2008) suggested that relatively large internal gaps might exist because of the bur size and the limited accuracy of scanning and milling. Consequently, the cement layer increases directly proportionally to the internal gap size. May, Kelly, Bottino, and Hill (2012) demonstrated through finite element analysis that the benefits of bonding to CAD/CAM crowns were lost as the cement thickness approached 450-500 μm due to polymerization thickness. Also through finite element analysis, Rekow, Harsono, Janal, Thompson and Zhang (2006) showed the stresses increase in glass-ceramic crowns with increased cement thickness. Furthermore, voids in the cement layer of the occlusal region present a significant potential mechanism for crown failure by increasing the tensile stress in the ceramic (Anusavice & Hojjatie, 1992).

INDICATIONS AND CONTRADINDICATIONS FOR LITHIUM DISILICATE

Lithium disilicate can be utilized for "thin" veneers (0.3 mm), minimally invasive inlays and onlays, partial and complete crowns, implant superstructures, and three-unit anterior/premolar fixed dental prostheses. Lithium-disilicate restorations can be

monolithic or can be cut back to be veneered with a nano-fluorapatite glass-ceramic for enhanced esthetics (Schweiger, Frank, & Drescher, 1999).

Although lithium disilicate restorations are extremely versatile, there are certain limitations of the material. While they can be used for three-unit fixed dental prostheses in the anterior and premolar areas, four-or-more-unit fixed dental prostheses are contraindicated. Other contraindications, consistent with those for other ceramic materials, include parafunction of the patient, insufficient tooth structure, inadequate reduction, and the geometry of the preparation design (Silva & colleagues, 2012).

ADVANTAGES AND DISADVANTAGES OF LITHIUM DISILICATE

The physical composition of lithium disilicate contributes to its high flexural strength, ability to limit crack propagation, polishability, and optical properties. Lithium disilicate exhibits high flexural strength. IPS e.max® CAD and IPS e.max® Press have biaxial flexural strengths of 360 ± 60 megapascals (Fischer, Bühlenr-Zemp, & Völkel, 2009) and 400 ± 40 megapascals (Bühler-Zemp, Völkel, & Fischer, 2011), respectively, which is about two and a half times that of any other available glass-ceramic CAD/CAM dental restoration (D. Fasbinder & Dennison, 2010).

Translucency of ceramics such as lithium disilicate can be accomplished by closely matching the refractive indices of the crystals and the glassy matrix (Denry, 1996). To achieve higher opacity, the glassy matrix can be filled with other materials, such as aluminum oxide. These fillers augment the opacity of the ceramic by absorbing, reflecting, or refracting the light.

Whether the restoration is fabricated via press or CAD, the lithium disilicate is provided as a monolithic pre-manufactured ingot with few internal defects. Lithium

disilicate is composed of a highly dense, interlocking pattern of many elongated lithium disilicate crystals (≤ 6 μm x ≤ 1 μm) and secondary lithium orthophosphate crystals ($\leq$ 0.1 μm to ≤ 0.3 μm) (Etman, 2009). The small sizes and high density of the crystal contribute to the strength and polishability of the ceramic. As a result, a high brightness and high chroma can be simultaneously achieved (Bühler-Zemp, Völkel, & Fischer, 2011).

Clinically, a major shortcoming of all ceramics, including lithium disilicate, is that they are susceptible to fracture. Crack extensions appear to occur when the stored elastic energy (mechanical energy) released during extension exceeds the energy required to form new surfaces (surface energy) (J. R. Kelly, 1995); Lawn B, 1993). Surface flaws or cracks in the glass act as stress concentrators and govern the strength of the material and are, therefore, more critical than the same concentration of internal stress raisers (Campbell & Kelly, 1989). Bulk fracture, or catastrophic failure, of dental ceramic restorations has been well documented (K. Malament & Socransky, 2009); (D. J. Fasbinder, 2010); (Lawn & colleagues, 2004); (Thompson, Anusavice, Naman, & Morris, 1994). This can occur whether the restoration is monolithic or layered, but several more factors are involved with multilayered ceramics (Lawn & colleagues, 2004). Other variables involved with fractography of dental ceramics include the inherent material properties, occlusal loading modes, environment and magnitude of forces applied, and residual stresses or flaws within the material (Bhowmick & Meléndez-Martínez, 2007). Generally, the failure originates as a subsurface radial crack from the intaglio surface of the restoration where tensile stresses concentrate, then propagates to the cameo surface, causing catastrophic failure (Lawn & colleagues, 2004).

Besides its optical properties, lithium disilicates via press and CAD/CAM have higher fracture toughness (K_{IC}) values of 2 - 2.5MPa $m^{1\backslash2}$ and 2.5 - 3 MPa $m^{1\backslash2}$, respectively, than a conventional glass-ceramic of 1.2 – 1.4 MPa $m^{1\backslash2}$ (Wiedhahn, 2007). Fracture toughness indicates a material's resistance to crack propagation. Because dental ceramic restorations fail via crack growth from existing flaws, fracture toughness is an excellent predictive value of the amount of clinical stress a ceramic restoration can withstand before fracture (J. Kelly, 2004). In lithium disilicate, cracks propagate intragranularly, through the glassy matrix, because of the high-density microstructure of crosslinking crystals (60-70%). A reliable resin bond is possible through the ability to properly acid etch this glassy matrix to increase surface area, decrease surface free energy and leave exposure to the lithium disilicate crystals.

PHSYICAL PROPERTIES OF VENEERING PORCELAINS AND THE VENEER-CERAMIC INTERFACE

Veneering porcelains are designed to increase the esthetics of all-ceramic restorations. Optical effects such as opalescence, fluorescence, translucency, texture, value, hue, and chroma can be customized. However, due to the microstructure of veneering porcelains, physical properties such as biaxial flexural strength, fracture toughness, and Weibull modulus are inferior to conventional glass-ceramics. However, these physical properties are higher in a veneered glass-ceramic than in veneering porcelain alone. This phenomena may be explained by crystallization of the veneer layer, the increased toughness of the ceramic core, compressive residual stresses associated

with thermal expansion anisotropy, and compressive stress associated with the viscoelastic structural relaxation (Taskonak, Mecholsky, & Anusavice, 2005).

Taskonak and colleagues (2005) investigated these different theories for the increase of biaxial flexural strength of veneered bilayer lithium disilicate. X-ray diffraction analysis indicated the veneering ceramic consisted of primarily amorphous glass with an undetectable amount of fluorapatite crystals. Therefore, these authors concluded that the strengthening mechanism for the bilayer ceramics is not caused by crystallization of the veneer layer. Furthermore in this study, crack propagation in all specimens initiated at the surface of the veneer and continued through the veneer-core interface. Therefore, the increased toughness of the core compared with the veneer did not affect crack initiation or propagation. Because ceramic materials are sensitive to tensile stress, the coefficient of thermal expansion of the veneering porcelain must be lower than the more rigid framework. The maximum difference of coefficients of thermal expansion is suggested to be 0.4 (10^{-6} K^{-1}), which is calculated to be 13MPa (Taskonak et al., 2005). Tensile and compressive forces can develop because of different viscoelastic relaxation mechanisms in elastic-viscoelastic composites (Dehoff & Anusavice, 1989). However, these stresses vary linearly throughout various layers of the composite material. Therefore, the differences of coefficients of thermal expansion alone do not fully explain the differences between the veneered and the monolithic specimens. Although the resultant compressive stresses on the bilayer ceramic are a result due a combination of these phenomenons, tensile stresses are the primary cause for observed chipping.

PHYSICAL PROPERTIES OF RESIN CEMENT AND THE CEMENT-CERAMIC INTERFACE

The use of resin cements as an adhesive interface for ceramics can be justified by their low solubility, optical properties, high modulus of elasticity, high compressive strength, bonding quality, and resultant increased fracture strength of the adhered ceramic. Potential explanations for the increase of fracture strength include placing the ceramic under compression, diffusing stress across the ceramic interface, and resin infiltration into the ceramic flaws called 'crack-bridging' (Hill, 2007; Rosenstiel, Gupta, Van der Sluys, & Zimmerman, 1993).

Effective etching of the ceramic surface is considered an essential step for the clinical success of ceramic bonding. The surface of the ceramic, and thus the ceramic-resin bond is affected by variations in the concentration, time, and type of acid etchant (Della Bona, Anusavice, & Mecholsky, 2006). As an added benefit, Anusavice & Hojjatie (1992) theorize that the subsequent acid etching treatment of the ceramic reduces the stress concentrations at the flaw tips by changing the shape and direction of inherent flaws. For lithium disilicate, a 5% Hydrofluoric acid etch for 20 seconds is recommended by the manufacturer for lithium disilicate ceramics for an optimal surface characterization and increased bonding area of the ceramic surface prior to silanization.

The goal of silanization in ceramics is to form strong bonds across the surface. To accomplish this, the methacryl monomers, which contain a tialkoxy silane group first hydrolyze, and the intermediate product reacts with the hydroxyl groups of the

silicate surface of the glass-ceramics forming in a condensation reaction to form covalent bonds (Volkel, 2002). The end result is a hydrophobic ceramic surface primed for reliable resin bonding.

TENSILE TESTING METHODS OF BILALYERED AND MULTILAYERED CERAMICS

The microtensile bond strength test has been considered the most effective mechanical test to study bond strength in dentistry since these fractures tend to occur in the adhesion zone (Pashley et al., 1999). Although this method is more labor intensive than conventional tensile testing methods, it provides insight into the long-term durability of the resin-substrate bond. However, to date, no standardized, methodological guidelines have been established so caution must be taken when comparing results from different studies (Castro, Sadek, Batitucci, & Miranda, 2014).

The microtensile test measures the tensile force to separate two substrates bonded together with a luting interface. Generally, the two surfaces are bonded together and then sectioned with a cutting disk to produce multiple, smaller surfaces. This cutting method, however, may generate undesirable flaws, stresses, and torque forces of the specimens at the junction of the resin-substrate interface resulting in pre-test failures and possibly inconsistencies of the groups tested (Marocho, Ozcan, Amaral, Bottino, & Valandro, 2013). Additionally, the cutting speed and disk type has been shown to alter the microtensile bond strength of ceramic specimens to resin cement (Castro et al., 2014).

The development of the microtensile test was to help eliminate non-uniform stress distribution at the adhesive interface and to minimize the influence of interfacial defects of a composite to dentin and enamel due to geometric irregularities of organic tooth structure (Sano & colleagues, 1994). However, the microtensile test produces variable fracture-surface morphology and fracture origins for the same adhesive interfaces within the adhesion zone, so a careful interpretation of the failure mode is required to prevent inappropriate conclusions about the results of the microtensile test and the adhesion zone phenomenon (Della Bona & colleagues, 2006).

Failure types can be defined as adhesive (between layers), cohesive (within a layer), or a combination of the two. Ideally, mechanical bond testing should results in consistent adhesive failures with a low standard of deviation. Although commonly used and easily performed, conventional shear testing tends to produce unfavorable cohesive failures. Finite element analysis has shown that shear testing grossly underestimates bond strengths, affects the substrate more than the bonded surface, and are not recommended for clinical application of resistance to fracture (DeHoff, Anusavice, & Wang, 1995).

Tensile bond strength is inversely affected by the bonded surface area and is determined by dividing the failure load by the cross-sectional area of the bonded surface (Sano et al., 1994). According to Griffith's theory, it is more probable to find a surface flaw in a larger area than a smaller one. Since fractures initiate at the site of the flaw, the apparent tensile strength of the material decreases when the tested area increases (Escribano, Del-Nero, & de la Macorra, 2003). Therefore, a smaller bonded surface area will result in greater tensile bond strength (Escribano et al., 2003).

Recently, this test has further extended into measuring microtensile bond strength of resin to ceramics. Few studies have tested the tensile bond strength of resin to lithium disilicate (Castro et al., 2014); (Marocho & colleagues, 2013); (Dündar & colleagues, 2007); (Colares et al., 2013); (Guarda et al., 2012). To date, only one study has investigated the microtensile bond strength of resin cement to CAD/CAM manufactured lithium disilicate (Aboushelib, 2014). This study examined different surface treatments of three different types of lithium disilicate glass-ceramics and the resultant microtensile bond strength of a resin cement interface between these ceramics to a composite resin. To date, no studies have investigated the microtensile bond strength of a resin cement interface between two CAD\CAM manufactured lithium disilicate glass-ceramics. Due to the limitations of this area of study in the literature, further investigations on this topic are warranted.

SUMMARY

Lithium disilicate restorations have extremely promising physical and optical characteristics. These types of ceramics have approximately twice the fracture toughness of conventional glass-ceramics, while maintaining an optimal refractive index due to the physical characteristics of the glass and interlocking crystalline microstructure. Because of these characteristics, lithium disilicate offers a variety of restorative treatment options, ranging from veneers to three-unit fixed dental prostheses to multi-unit implant superstructures. Whether heat pressed or CAD/CAM manufactured, the versatility of

fabrication methods of lithium disilicate dental restorations benefit the dentist and the patient.

Numerous *in vitro* and short-term *in vivo* studies indicate that lithium disilicate dental restorations may prove groundbreaking in esthetic restorative dentistry. Because lithium disilicate is a relatively new dental ceramic, there are no long-term *in vivo* studies to verify their full potential. Whether the lithium disilicate restorations are monolithic or veneered, the fabrication processes can alter the physical characteristics of the ceramic and ultimately affect its optical and structural integrity. Thus, the tensile bond strength of a ceramic-resin-ceramic interface should be explored as an alternative for a restorative option. There appears to be a gap in the literature to compare the tensile bond strength of a high translucency CAD/CAM fabricated lithium disilicate glass-ceramic resin bonded to a low translucency CAD/CAM lithium disilicate glass-ceramic, so therefore the purpose of this study is to directly compare the tensile bond strength of this tri-layered glass-ceramic using three different resin cements *in vitro*.

MATERIALS AND METHODS

A standardized Dumb-bell test according to ISO 11405:2003(E) will be used to test and compare the tensile bond strength of two different resin cement interfaces of a low translucency lithium disilicate ceramic to a high translucency lithium disilicate ceramic. The two different resin cements to be tested: Variolink® 2 (Ivoclar Vivadent, Liechtenstein, Germany), and Panavia F 2.0® (Kuraray, Kurashiki, Japan). The testing will utilize ten dumbbell specimens for each respective group of resin cement (n=10 for each resin cement group) (Table 1).

	Group 1	Group 2
Half-dumbbell substrate 1	e.Max CAD HT A2	e.Max CAD HT A2
Resin Interface	Panavia F 2.0	Variolink 2
Half-dumbbell substrate 2	e.Max CAD LT A2	e.Max CAD LT A2

Table 1. Groups of tested interfaces under tensile force.

The half-dumbbell specimens were designed using SolidWorks 3D design software (Waltham, Massachusetts) with dimensions described in Figure 3.

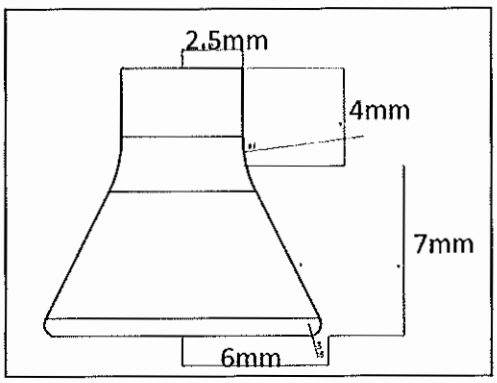

Figure 3. Schematic (CAD design of half dumbbell to include dimensions)

All specimens will be IPS e.max CAD® (Ivoclar Vivadent, Liechtenstein, Germany) lithium disilicate ceramics milled from a Ceramill Motion 2 (Amann-Girrbach, Charlotte, NC) 5-axis mill. All milled ceramic specimens will be crystalized according to manufacturer's specifications. A total of 20 half-dumbbell shaped ceramic specimens will be low translucency (LT) shade A2 lithium disilicate and 20 half-dumbbell shaped ceramic specimens will be high translucency (HT) A2 lithium disilicate (Figure 4).

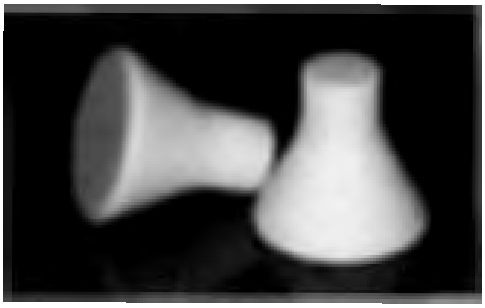

Figure 4. Pre-crystalized half-dumbbell specimens of lithium disilicate LT A2 and HT A2

The surface area of each half-dumbbell will be 19.6mm². This 19.6mm² surface will be acid etched according to manufacturer's instructions with IPS Etching Gel® (Ivoclar Vivadent, Liechtenstein, Germany), a 5% hydrofluoric acid (Figure 5), and allowed to react for 20 seconds, and rinsed copiously with water, subjected to steam cleaning, then allowed to dry. For the Variolink 2® group, a thin coat of Monobond Plus® (Ivoclar Vivadent, Liechtenstein, Germany) restorative primer will be applied and allowed to react for 60 seconds. For the Panavia 2.0 F® group, a thin coat of Clearfil Ceramic Primer (Kuraray, Kurashiki, Japan) will be applied according to manufacturer's specifications and allowed to dry. The respective resin cements will then be applied to the treated surfaces of each half-dumbbell specimen and placed in a standardized index

jig which incorporates a 0.10mm gap size between ceramics. The index jig will be additively manufactured from an Objet500 Connex (Stratasys Corporation, Eden Prairie, MN) 3D printer with a photopolymerizing resin (Figures 6, 7).

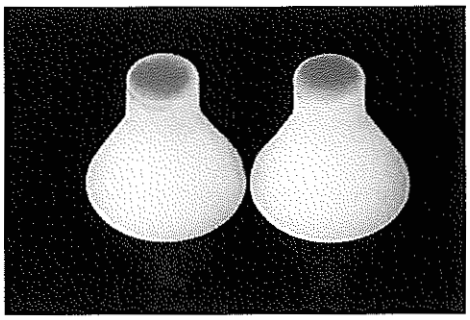

Figure 5. LT and HT half-dumbbell specimens with 5% Hydrofluoric acid etch.

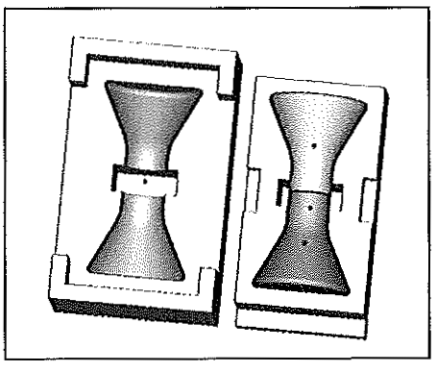

Figure 6. CAD design of specimen seating jig with design of two half-dumbbell specimens inserted.

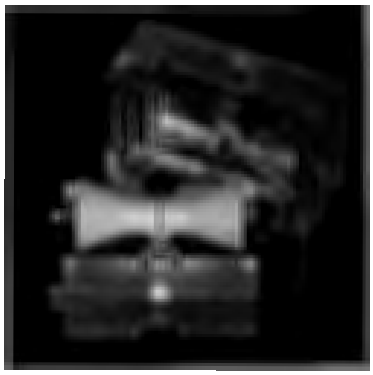

Figure 7. Photo of LT and HT half-dumbbells in indexing jig with 0.10mm gap between crystalized specimens

After full polymerization and cleaning of excess cement, the dumbbell specimens will be placed in a MTS Insight 5kN load cell with customized grips and placed under tension at a crosshead speed of 0.5mm/min until the resin interface is broken (Figure 8). Data collected will consist of load, displacement, stress, and strain.

Figure 8. MTS Insight 5kN load cell with resin cemented specimens under tensile force.

RESULTS

Results from the test are listed on Table 2 and Figures 9, 10, 11, 12.

		Mean (MPa)	Std. Deviation
Peak Stress (MPa)	Panavia F 2.0	9.77	5.57
	Variolink 2	12.15	9.17
Strain at Break (%)	Panavia F 2.0	4.06	5.46
	Variolink 2	1.99	0.89
Modulus of Elasticity (GPa)	Panavia F 2.0	0.51	0.27
	Variolink 2	0.62	0.37
Peak Load (N)	Panavia F 2.0	184.07	113.11
	Variolink 2	238.42	180.47

Table 2. Resulting means and standard of deviations from the Panavai F 2.0 and Variolink 2 groups.

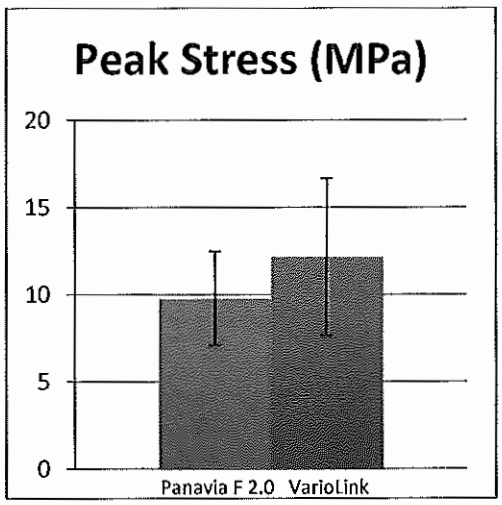

Figure 9. Mean Peak Stress (MPa) with standard of deviation.

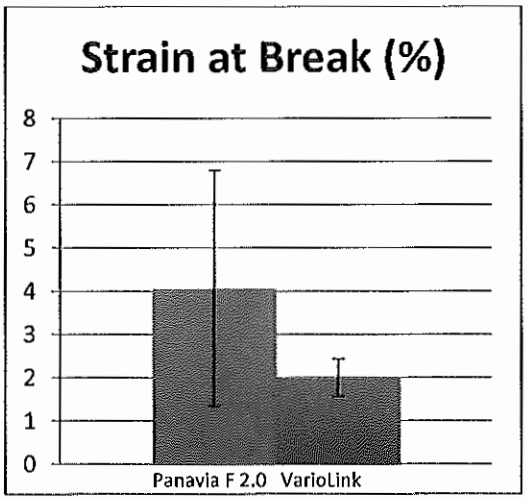

Figure 10. Mean Strain at Break (%) with standard of deviation.

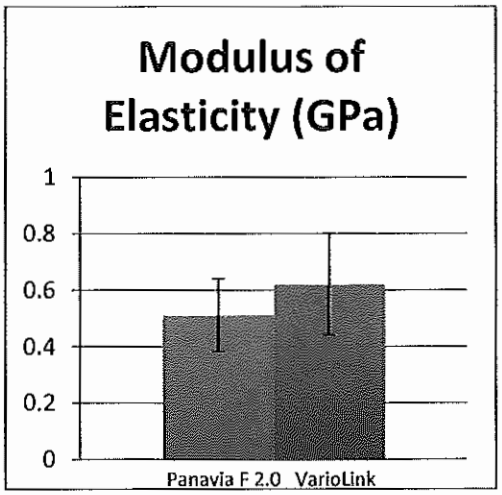

Figure 11. Mean Modulus of Elasticity (GPa) with standard of deviation.

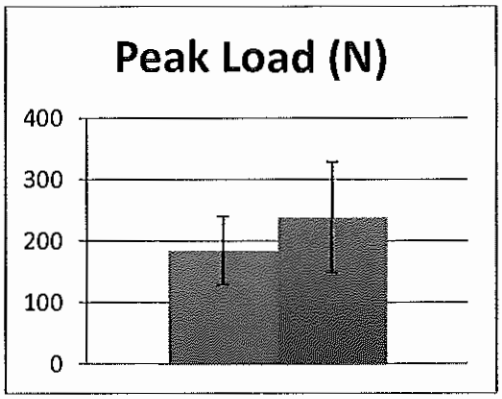

Figure 12. Mean Peak Load (N) with standard of deviation.

Due to the limitations related to sample size used in this study (n=10), Levene's test of homogeneity of variables was used which indicated that equal variances for each sample could not be assumed for Peak Stress, Modulus of Elasticity, and Peak Load. However, the variances were homogenous for the Stain at Break (P = 0.038) (Table 3). Since this assumption could not be assumed for each group, the Mann-Whitney U Test tested the independent samples. This test confirmed that the Panavia F 2.0 and Variolink 2 groups were not statistically different within each independent variables being Peak Load (p = 0.739), Peak Stress (p = 0.853), Strain at Break (p = 0.315), Modulus of Elasticity (p = 0.436).

	Levene's Test for Equality of Variances		t-test for Equality of Mean	
	F	Sig.	t	df
Peak Stress (MPa)			-0.701	18
	2.5	0.131	-0.701	14.846
Strain at Break (%)			1.178	18
	5.042	0.038	1.178	9.479
Modulus of Elasticity (GPa)			-0.792	18
	2.402	0.139	-0.792	16.662
Peak Load (N)			-0.807	18
	2.076	0.167	-0.807	15.126

Table 3. Levene's Test for Equality of Variances

DISCUSSION

The results of the present investigation cannot justify the rejection of the null hypothesis of a difference between the tensile bond strength of Panavia F2.0 and Variolink 2 as an interface between these a high translucency and low translucency lithium disilicate glass-ceramics due to no statistical difference between the two groups was found. Panava F 2.0 and Variolink 2 are two commonly used dual curing resin cements. Both products claim impressive bonding ability to tooth structure, ceramics, and metal alloys, however Variolink 2 has more esthetic options as well as try-in pastes. Another major difference between the two products is that Panava F 2.0 has separate primers for ceramics and metals, whereas the primer for Variolink 2 can be applied to both ceramics and metals. Any differences in optical properties, ease of use, and bonding capability can have an impact on clinical decision making for product selection.

After testing, the first samples from the Panavia F 2.0 and Variolink 2 groups were reviewed under the Hyrox microscope to analyze for adhesive and cohesive failures. Observation of the Panavia sample demonstrated a mix of ceramic and residual resin cement surface suggesting a mixed adhesive-cohesive failure of the resin-ceramic interface (Figures 15, 16). The Variolink sample also demonstrated a mixed ceramic and residual cement surface suggesting a mixed adhesive-cohesive failure of the resin-ceramic interface (Figures 17, 18).

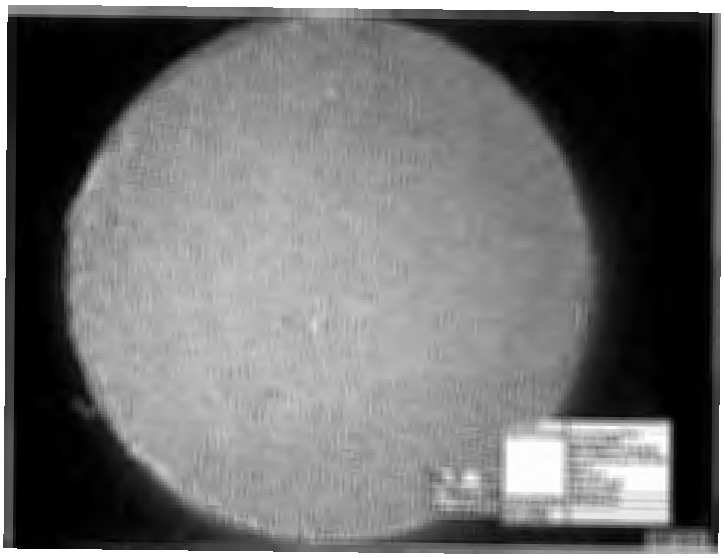

Figure 13. Panavia 2.0 High Translucency specimen under microscope demonstrating etched ceramic surface and residual adhered resin cement.

Figure 14. Panavia 2.0 High Translucency specimen under higher magnification showing the residual adhered resin cement (top) and etched ceramic surfaces (bottom)

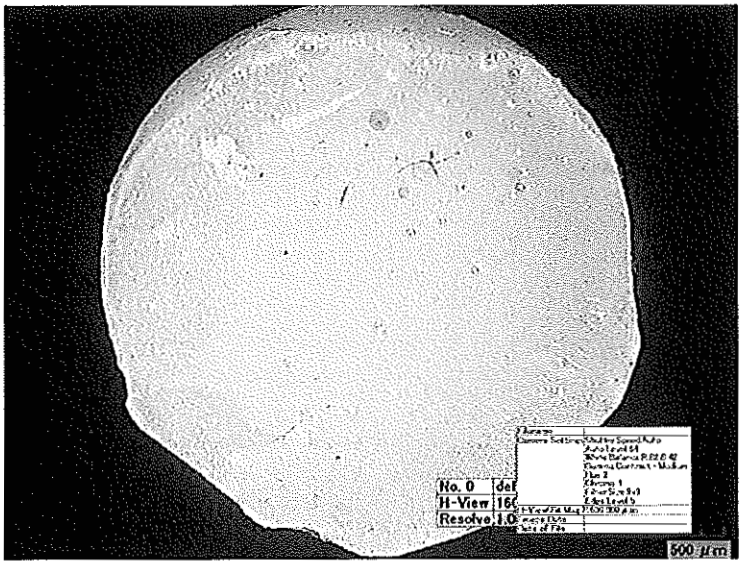

Figure 15. Variolink 2 High Translucency specimen under microscope demonstrating etched ceramic surface and residual adhered resin cement.

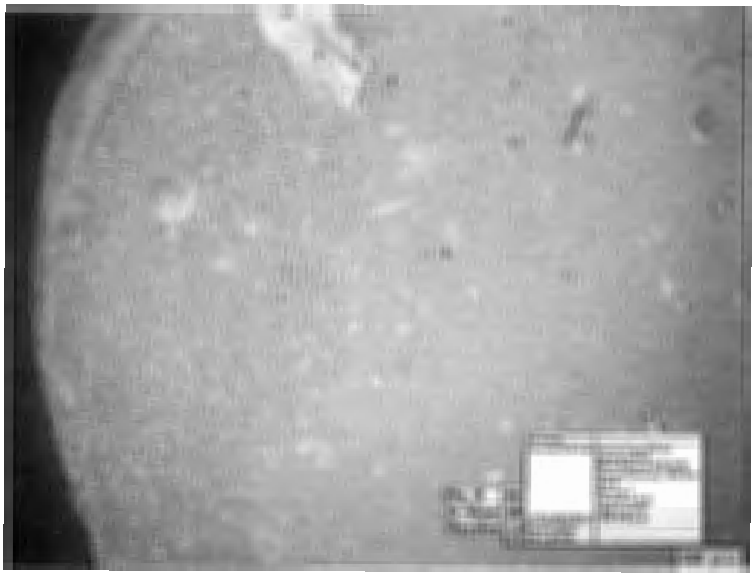

Figure 16. Variolink 2 High Translucency specimen under higher magnification showing the residual adhered resin cement (right) and etched ceramic surfaces (left)

One source of error which likely had an impact on this study was the human error associated with removal of the sprue from the ceramic specimens. Due to the nature of subtractive manufacturing, a support sprue must be incorporated into the design to stabilize the specimen during the milling process (Figure 13). After the specimens were milled, the support sprues needed to be manually removed with hand instrumentation. This lack of uniformity may have had an impact on the positioning of the specimens in

the cementation jigs during resin cementation and also may have also produced inconsistent positioning in the titanium grips during tensile load testing.

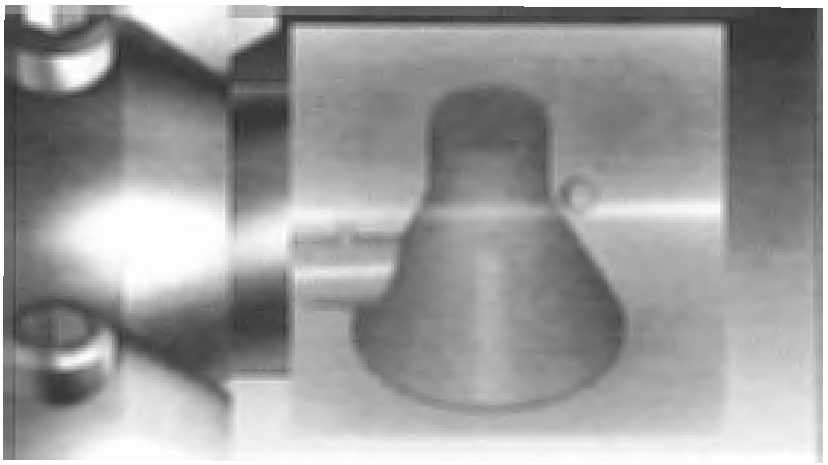

Figure 17. Half dumbbell specimen in virtual design showing sprue attachment

This theory is supported by the multiple failure points seen from specimen #5 from the Panavia group and specimen's #'s 8 and 10 from the Variolink group. Multiple fractures were seen in the ceramic specimens at a distance from the resin interface and likely where the ceramic contacted the titanium grips (Figures 14, 15).

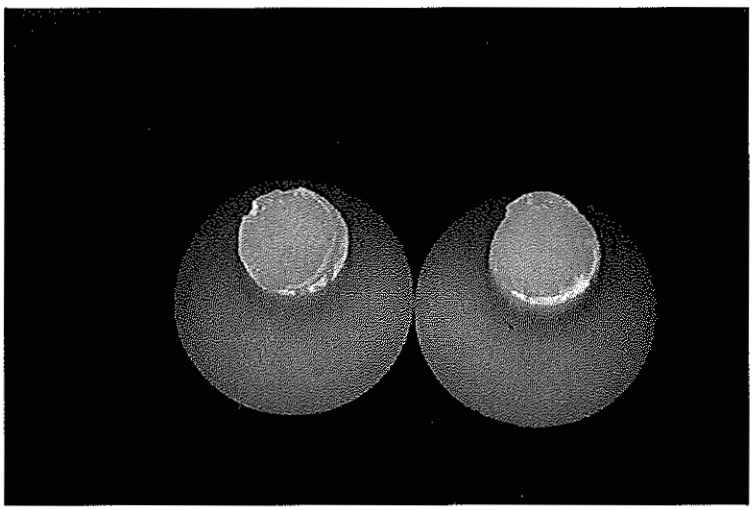

Figure 18. Panavia F 2.0 specimen #6 demonstrating desired typical pattern of failure at the resin-ceramic interface

Figure 19. Variolink 2 specimen #8 demonstrating multiple undesired ceramic fractures at a distance from the resin cement interface

CONCLUSIONS

Because of the promising physical and optical properties of lithium disilicate, the ceramic-resin-ceramic interface can be a promising restorative option for the restorative dentist. Clinical examples of this interface are the ceramic customized abutment resin-bonded to a ceramic crown as well as a ceramic veneer overlay resin-bonded to a ceramic core substrate. Although there appears to be a gap in the literature that explores this interface, this in vitro study does not reject the difference in tensile bond strength between two commonly use dual-cured resin cements.

Under the conditions of this investigation the following conclusion can be drawn: There is no statistical significance in tensile bond strength in regards to Peak Load, Peak Stress, Strain at Break, and Modulus of Elasticity between Panavia F 2.0 and Variolink 2 as a resin cement interface between a high translucency and low translucency lithium disilicate glass-ceramics. Differences exist between these two products such as ease of application and optical properties. Variolink 2 has one silane primer for the both metal and ceramics while Panavia F 2.0 has two different silane primers. Variolink 2 is also less opaque and provides more esthetic options than Panavia F 2.0. The findings of this study in conjunction with these clinically relevant examples may lead the clinician to choose a resin cement for the ceramic-resin-ceramic interface for reasons other than tensile bond strength. Future studies should continue to explore the bond potential, physical properties, and clinical implications of the ceramic-resin-ceramic interface.

APPENDIX

SPECIMEN COLLECTION DATA SHEET

Panavia F 2.0

Spcmn No.	Diameter mm	Peak Load N	Peak Stress MPa	Strain At Break %	Modulus GPa
1	5	303.935	15.5	2.063	0.749
2	5	82.223	4.2	2.362	0.178
3	5	198.395	10.1	1.417	0.713
4	5	117.274	6	1.901	0.396
5	5	440.646	22.4	6.512	1.028
6	5	168.416	8.6	1.756	0.488
7	5	161.173	8.2	1.195	0.687
8	5	104.793	5.3	1.807	0.295
9	5	71.023	3.6	2.411	0.153
10	5	192.953	9.8	2.107	0.477

Table 4. Data collected from Panavia F 2.0 group

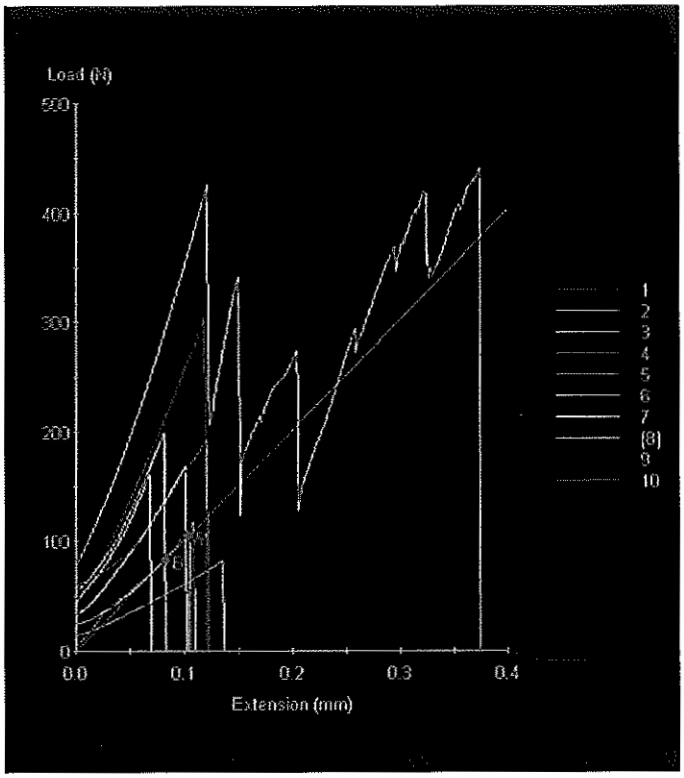

Figure 20. Data collected from Panavia F 2.0 specimens 1-10

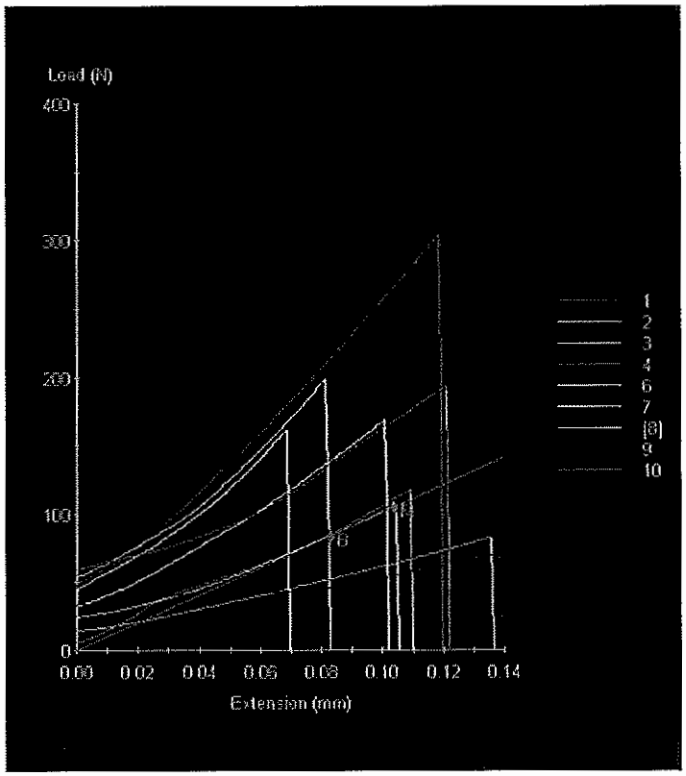

Figure 21. Data collected from all Panavia F 2.0 specimens excluding specimen #5

VarioLink 2

Spcmn No.	Diameter mm	Peak Load N	Peak Stress MPa	Strain At Break %	Modulus GPa
1	5	170.987	8.7	1.684	0.526
2	5	372.661	19	1.944	0.976
3	5	93.295	4.8	1.485	0.32
4	5	60.824	3.1	2.439	0.127
5	5	249.606	12.7	1.628	0.781
6	5	65.832	3.4	1.552	0.216
7	5	239.894	12.2	1.489	0.819
8	5	613.485	31.2	4.409	1.036
9	5	106.275	5.4	1.563	0.348
10	5	411.387	21	1.847	1.16

Table 5. Data collected from Variolink 2 group

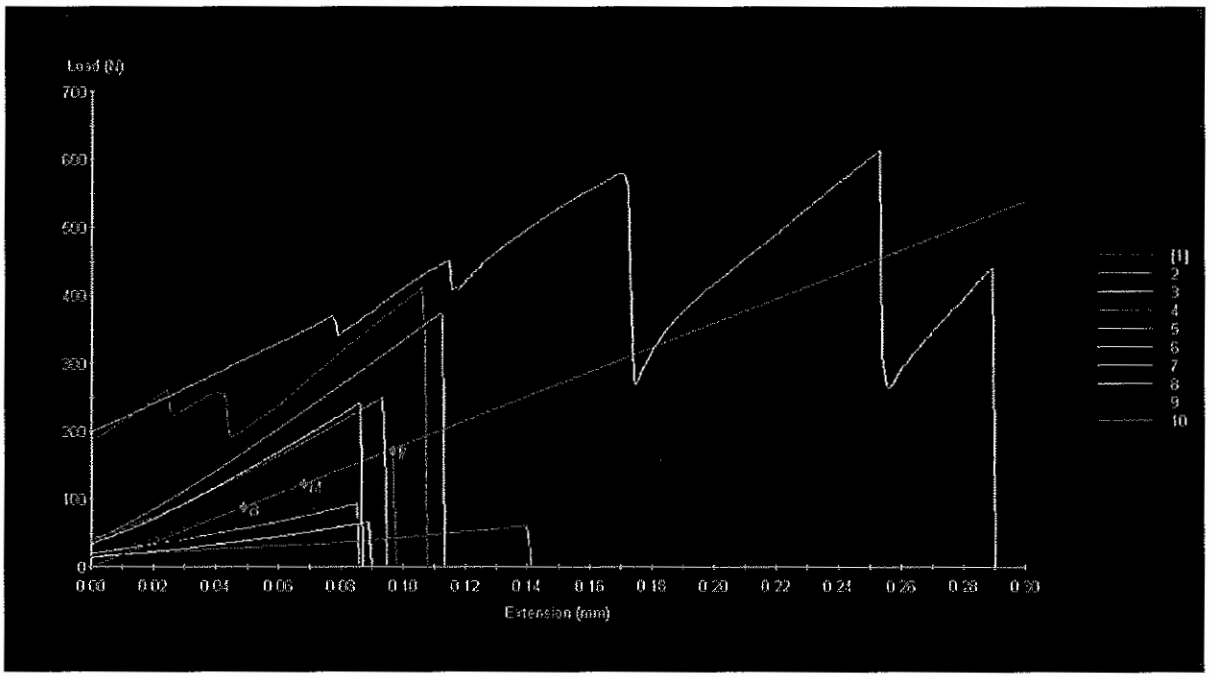

Figure 22. Data collected from Variolink 2 specimens 1-10

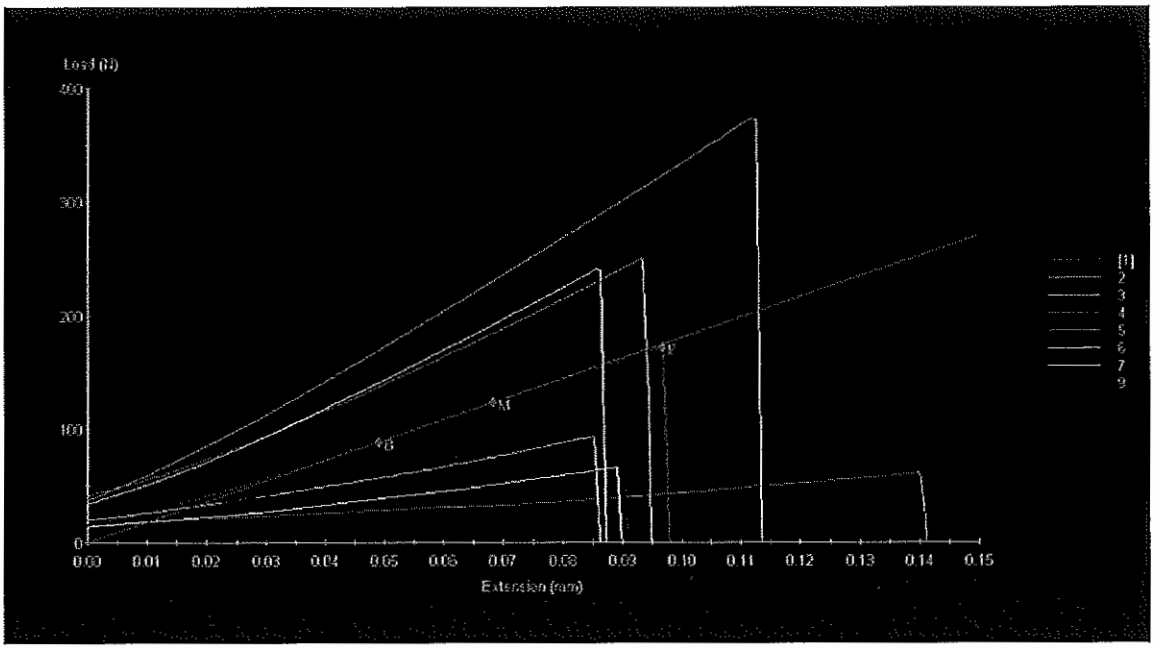

Figure 23. Data collected from all Variolink 2 specimens excluding specimens #8 and #10

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