Analysis of Flexural Strength and Monotonic Load to Failure Following Simulated Chairside Adjustments and Repair in a Lithium Disilicate Glass-Ceramic

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Analysis of flexural strength and monotonic load to failure following simulated chairside adjustments in a lithium disilicate glass-ceramic

By

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ABSTRACT

ANALYSIS OF FLEXURAL STRENGTH AND MONOTONIC LOAD TO FALIURE FOLLOWING SIMULATED CHAIRSIDE ADJUSTMENTS IN LITHIUM DISILICATE GLASS-CERAMIC

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Introduction: There are no studies regarding e.max Press that evaluate post-adjustment healing protocols and their effect on the load to failure in a clinically relevant test. It is essential to find the ultimate treatment protocol which will help clinicians preserve the physical properties of the ceramic restoration after adjustment.

Material and Methods: The total number of samples used in this study was 440 IPS e.max press discs. The discs were 15 mm in diameter and 1 mm of thickness. The material was tested for flexural strength and monotonic load to failure. The test was done in two parts and with the same tests. The flexural strength contained 40 specimens per group while the load to failure group had 20 specimens per group. In the biaxial flexural test, the specimens were loaded at 0.5 mm/min until failure using a ring on ring arrangement and the biaxial strength was recorded. The monotonic load to failure specimens were cemented with a resin cement on epoxy resin blocks, and loaded with a 50-mm hemisphere at a cross head speed of 0.5 mm/min. The tests were performed on a universal testing machine (Instron). Weibull statistics determined intergroup differences.

Results: In Part I: regarding the flexural strength tests, the Weibull plot and likelihood ratio contour plot revealed a significant difference between the control group and the other groups. Regarding the monotonic load to failure tests, the Weibull plot and likelihood ratio contour plot revealed no significant difference between the control and glazed groups. The diamond adjusted group was significantly different from the control group and the glazed group.

In Part II: Regarding the monotonic load to failure, the Weibull plot and likelihood ratio contour plot revealed no significant difference between the tested groups. The strength of all the groups when subjected to glaze treatment after divesting increased in comparison with groups in Part I.

Conclusions: Glazing treatment improved the physical properties of adjusted IPS e.max Press discs when subjected to biaxial flexural test and monotonic load to failure.
When clinical adjustments are made on the IPS e.max Press intaglio surface, a subsequent glazing treatment is recommended.

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CHAPTER 1

INTRODUCTION

Selection for lithium disilicate glass-ceramic (LDGC) restorations have increased significantly in the field of prosthetic dentistry.[1] The material possesses remarkable beauty and strength and is composed of an acicular crystalline material (70%) embedded in a glassy matrix. The translucency, esthetics, and successful clinical performance have made LDGC one of the most popular all-ceramic materials.[1,2] With the increased demand for metal-free restorations, LDGC meets the requirement for a material possessing strong mechanical properties combined with the optical properties of natural teeth.[1,2] Mechanical strength is one of the main factors that determines the clinical success of all-ceramic restorations. In vitro studies report flexural strength of 360-440 MPa and fracture toughness of 2.25-2.75 $MPa.m^{0.5}$.[3,4] These numbers are low when compared to zirconia or even alumina; however, the performance of glass-ceramic should increase after etching, silanization and bonding to prepared dentin using an adhesive resin cement.[5] The mechanical and physical properties of the material allow it to be used in various applications ranging from inlay/onlay, single complete crown and short fixed dental prostheses.[4,6] Clinical performance data show survival rates up to 97.6% after 5 years; however, complications have also been reported.[6]

In a clinical situation, chairside adjustments are frequently necessary to improve seating and marginal fit of a prosthesis. Moreover, adjustment of the cameo or the occlusal surface is often performed to improve occlusion.[7,8] Studies have demonstrated that cracks and flaws will form in lithium disilicate materials following chairside adjustment with a diamond rotary cutting instrument or in the research laboratory following indentation procedures.[9] Cracks and flaws may also initiate during milling procedures or after etching a restoration with hydrofluoric acid.[10] Subsurface cracks and flaws have been determined to be the main cause for failure of ceramic restorations.[11] Clinical testing and retrieval analysis have demonstrated that all-ceramic failure most often originates from the cementation surface.[12] It is believed that during mastication, occlusal forces will create a tensile stress at the cementation interface. Once the stress reaches a critical level the restoration will fail as result of uncontrolled crack growth.[13] Because lithium disilicate is a glass-ceramic, it is susceptible to another process called slow crack growth. In this process, the stress is subcritical and may lead to restoration failure in the presence of moisture and over time.[14]

Hydrofluoric acid has been shown to increase the surface roughness and consequently weaken LDGC.[15] However, when LDGC is bonded to a tooth using resin cement, the strength of the restoration was unaffected by etching.[16] In another study, heat treatment, glazing and veneering following abrasive grinding of the internal surface of LDGC healed the cracks and the defects.[9] A simple polishing protocol was shown to be effective in smoothing cracks and resulted in an increased fracture load.[17] These techniques of healing the cracks and flaws have not been directly compared in a single study. It is essential to find the ultimate treatment protocol which will help clinicians preserve the physical properties of the ceramic restoration after adjustment.

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Historic Background

Early in the 18th century, missing teeth were replaced with animal products or extracted teeth from dead bodies.[18] The Chinese were the first to master production of hard translucent porcelain in the 200s.[18] Ceramic materials have been used as early as AD 300 as restorative material.[19] Archaeologist's found teeth from the Mayan period decorated or restored with ceramic and it was believed to be for esthetic reasons.[19] In the late 1600s porcelain become a hot research topic in Europe. In 1770, the pharmacist Alexis Duchateau was the first to attempt replacement of teeth with porcelain dentures.[20,21] Porcelain shrinkage and malodor were problems for him.[20,21] He then sought the help of Nicholas Dubois De Chemant, a Parisian dentist, to overcome the shrinkage problem and together they succeeded. De Chemant then moved to England and he altered a few components in the porcelain formulation which resulted in a product close to the feldspathic porcelain that we have nowadays.[20, 21] Duchateau invented the process but De Chemant perfected the recipe. [20, 21]

In 1806 Giuseppangelo Fonzi an Italian dentist was able to produce full contoured individual porcelain teeth containing a platinum pin.[22] It was a great advancement as it allowed setting teeth on metal-frameworks and repair was made easier.[22] In 1895 Charles Henry Land developed fabrication of porcelain crowns with a platinum matrix.[23] Platinum was heavily used in that era as it has a thermal contraction close to porcelain.[23] Land made the first laminate porcelain veneer in 1901.[23]

In 1965 Mclean and Hughes made a great advancement in the dental ceramic field.[24] They developed a porcelain core that contained aluminum oxide particles.[24] The alumina cores have a coefficient of thermal expansion matching the veneering porcelain that was baked on it.[24] The flexural strength recorded was 180 MPa which was twice as much as conventional feldspathic porcelain.[24]

In 1980, Dicor became the first glass-ceramic commercially available. It is mica based and is considered a castable ceramic (processed with lost wax technique).[25,26] In 1987 the first scanned restoration was marketed (CEREC1, Sirona). Mormann and Brandestini used a machine to scan a prepared tooth and fabricate a 3D restoration chair-side using computer-aided design software and computer-assisted manufacturing (CAD/CAM).[27] University of Zurich reported pressing leucite glass-ceramic (IPS Empress) in 1990.[26] The product was marketed as a bonded all-ceramic restoration. It has superior esthetics and a strength of 180 MPa.[26] Lithium disilicate glass ceramic was first created in 1998 by Ivoclar. Ivoclar improved the strength of the material by increasing the crystalline content and refining the particle size which changed the microstructure. [27]

Modern Dental Ceramics

There are three main classes of dental ceramics based on their microstructure.[28] Glassy microstructures (feldspathic), partially crystalline glass-ceramics and polycrystalline ceramics. Ceramics with a predominant glassy microstructure are more esthetic while ceramics with more crystalline phase are stronger.

Ceramics with a predominantly glassy phase are composed of matrix (alumina-silicate glass) and filler (colorant and opacifiers).[29] Their main use is to veneer substructures and they possess different filler composition for different substructures.[29] Because of their irregular amorphous microstructure, optical properties are excellent. Ceramics with a partially crystalline content possess greater strength.[27,30] The crystalline phase determines the physical properties of the product.[27,30] The fabrication process of these materials may involve pressing or CAD/CAM procedures.[27,29] The most popular material in this category are leucite-based glass-ceramics with a 40% crystalline content or lithium disilicate glass-ceramics with a 70% crystalline content.[27,30]

Polycrystalline ceramics have no glass content. The most popular products from this category are composed of alumina or zirconia. They can be fabricated with CAD/CAM technology.[27,30]

Lithium Disilicate

This all-ceramic system was initially introduced by Ivoclar as Empress II. Now it is in the form of IPS e.max. The microstructure is made of lithium silicate glass matrix embedded with lithium disilicate crystals. Refining the crystal size and increasing the crystal content are two improvements incorporated into the present IPS e.max product.[31] IPS e.max is available for pressing, as well as, for machining with CAD/CAM technology. The final product can be delivered as a layered restoration or as a monolith. A fluorapatite is used to veneer the e.max core and create the final shape and shade.[31] The low refractory index of the lithium disilicate crystals is responsible for the translucency and pleasing optical properties.[32]

Strength is defined as the stress that a material can withstand before it breaks, or the load applied per unit area. IPS e.max has a flexural strength of 400 MPa.[3] One major reason for failure of ceramic restorations are surface flaws. Flaws in a ceramic can be inherent in the microstructure or introduced during machining or clinical adjustments.[9, 11, 32] Crack propagation increases as the applied load increases. Clinical studies reported that ceramic strength depends upon the elastic modulus of the coping or abutment, the thickness of the restoration, thickness and quality of the cement and loaded contact area.[33]

Fracture toughness is another physical property that measure a materials resistance to crack propagation.[34] It is an inherent property of the material[32,34], that should be considered when designing and selecting a restoration for use in the mouth.[35]

Glass-ceramics

Most glass-ceramics are fabricated by a process called ceramming. It is a controlled heating process that will convert the non-crystalline microstructure of glass ceramic into a crystalline microstructure. This process takes place in two phases, crystal nucleation and then crystal growth. Formation of the crystalline phase in the glassy matrix increases strength. The crystalline phase will interrupt crack propagation.

Clinical Failure of Ceramic Restorations

Fracture surface analysis and fractography of clinically failed Dicor crowns revealed that a crack initiated in the intaglio surface of the restoration and propagated to the cameo surface resulting in bulk fracture.[12,13, 36] Under functional forces the ceramic will exhibit a tensile stress on the cementation surface. This state will lead to minor bending in the ceramic layer. Under functional loads, the ceramic will develop more tensile stress and if there is a crack or a flaw it may propagate in a subcritical manner until it reaches a point where it results in catastrophic failure. Degradation of the bonding agent is another factor that can contribute to bulk fracture. [37] As the cement degrades and begins to leak, slow crack growth may assist in the catastrophic failure.[38] Most laboratory studies fail to replicate clinical failure and resulted in damage on the cameo surface leading to the bulk fracture.[39,40,41] Modern glass-ceramics are believed to fracture similarly, and the margins are identified as the weakest point of the crown and where fracture initiates.[42,43] The crack will propagate parallel to the walls and then lead to a horizontal split.

Origin of Defect

The presence of a crack in ceramic restorations may limit the clinical performance, as it will make the restoration vulnerable to failure. All-ceramic restoration are fabricated by different techniques and each technique produces different flaws with respect to geometry and distribution.

A. Fabrication technique

Heat pressing

The pressing fabrication technique was found to lead to porosity formation in the final product. An in vivo study by Guazzato et al[44] found that pressed lithium disilicate samples exhibited 3% porosity and leucite-based exhibited almost 10% porosity in the final product. The reaction with the phosphate-bonded investment material will result in the formation of a reactionary layer. Oftentimes air abrasion and grinding are necessary to remove the attached reactionary layer. These laboratory steps may create cracks and flaws[9,11] which negatively impacts the long-term performance of the restoration. In response to a load, stress concentrations will increase around the flaw and crack propagation may initiate.

CAD/CAM

Cracks can result during the production phase in the CAD/CAM machining process.[45] An in vitro study of 400 samples of ceramic; machining defects (cracks) were reported and it was found that cracks were strongly associated with the grit size of instrument.[46] With lithium disilicate the restoration is fabricated prior to the full crystallization stage and is then subjected to heat treatment to complete crystallization. It was noticed that heat treatment reduced residual stress but machining damage was not eliminated.

B. Chair-side occlusal adjustment

It is common practice to improve restoration seat, marginal adaptation or adjusting the occlusion with a diamond rotary cutting instrument or adjusting kit. It is reported that these clinical steps will create cracks and flaws. In a laboratory study, surface defects have been shown to negatively impact strength of the restorations where adjustment with a diamond rotary cutting instrument was performed. . The data showed a reduction in the strength compared with a non-adjusted control because of the adjustment.[9,41]

Ruschel et al 2014 conducted a study to evaluate the effect of external and internal adjustment with and without a polishing procedure on the flexural strength of lithium disilicate specimens. [47] One group received a glazing treatment while a no-polishing group was used as a control. The specimens were adjusted with fine diamond rotary cutting instruments positioned perpendicular to the specimens. The depth of the adjustment was not mentioned. Specimens were tested with a 3-point bend test and the strength was 200 MPa lower than previous reports (400 MPa). There was no significant difference between the polishing protocols and the control.

Another study conducted by Hung et al 2008, evaluated the effect of diamond grinding on the intaglio surface of a ceramic restoration to improve fit. [9] The group proposed six different methods to heal the diamond adjustments. The depth of adjustment was not reported. Specimens were tested in a biaxial flexure setup using a three-ball-on-ring arrangement. The group found that veneering after pressing and divesting increased the strength. Diamond grinding reduced the strength of the specimens. In addition, subsequent heat treatment from veneer firing or glazing improved the strength.

Prevalence of bulk fracture of lithium disilicate

There is an increasing number of clinical studies reporting on the longevity and success of ceramic restorations. They precisely describe the number and nature of failed restorations from the day of insertion. One clinical study, evaluated over a period of 10 years, observed 261 IPS Empress II restorations, it was found that only 0.8% of the failures could be attributed to the restorations and it was at the 48 and 75 months post insertion.[48] Another clinical study reporting on the clinical performance of IPS e.max press over a period of 9 years and found that among 94 samples, 3.3% exhibited minor chipping, at 6, 31 and 92.6 months, but only 2.1% of the crowns exhibited bulk fracture and it was at the 92.6 and 101.2 months. [49]

Here, one can ask a fair question as to how and when does the damage affect the clinical performance?

It is very important to understand the mechanism of failure in ceramic restorations and to have a laboratory tests that can simulate the clinical condition and promote failure similar to those reported clinically. It is also essential that the magnitude of failure loads are equal or in close range to forces of mastication. This could help in studying defect size and relate it to clinical performance. Also, it would allow determination of what is a permissible defect size that promotes normal performance. Moreover, it will open doors to creating protocols to heal and restore defects.

CHAPTER II

BACKGROUND AND SIGNIFICANCE

Laboratory mechanical testing methods that stimulate relevant clinical fracture

For a laboratory test to mimic the clinical situation it must replicate clinical variables. Failure loads should be within a clinically reported range and the pressure contact area should not exceed what would resemble the clinical situation. Also, the test should produce cracks that have the same behavior as a clinical crack. Dr. J. Robert Kelly at the University of Connecticut developed a testing protocol that resulted in laboratory failure similar to what has been reported clinically. [33]

About the mode of failure of dental ceramic in laboratory studies and compare it to the clinical observations.

In laboratory studies, the contact between the spherical indenter and a ceramic specimen can be best described as a non-conformal contact. [50] A non-conformal contact is a small contact area with high stress. When the spherical indenter comes into contact with a flat or occlusal surface of a specimen, the real contact area is the sum of the asperities. As the load increases the real contact area increases (Figures 1a and 1b). [50]

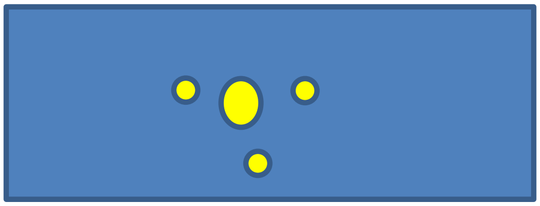

Figure 1a
Schematic diagram of spherical surface contact with flat surface (Initial contact).

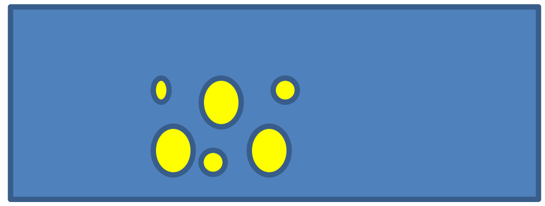

Figure 1b
Schematic diagram of spherical surface contact with flat surface. (As the load increase).

Two types of deformities can arise at that point, elastic and plastic. When the two surfaces come into contact, the maximum stress is some distance below the Hertzian contact pressure which is described as Von Mises effective stress. [50] When friction increases between the two surfaces, the stress moves upward towards the surface. [50] A cone crack or median crack results when the Hertzian contact pressure is high and leads to crushing damage. This type of failure has only been observed in laboratory studies. In the clinical situation, a radial crack initiates from the cementation layer and extends to the occlusal surface and results in a cone crack.

Testing physical properties:

In the oral cavity, the dental restoration is subjected to different types of forces; tensile, compressive, shearing and torsional. The most common type of force is bending forces which is a combination of compressive and tensile force vectors. The elastic modulus is a useful property for assessing mechanical properties of a dental material. Elastic modulus is a property defined by the stress (force per unit area) over strain (deformation of the material). When a plot of a stress-strain curve is made, important features of a material can be calculated. The first feature is the stiffness of the material or Young's modulus of elasticity which can be calculated from the slope on the stress strain graph. Secondly, the maximum stress or the proportional limit of the material is where a plastic deformation will occur after exceeding this limit. The yield strength is similar to the proportional and is typically measured at 0.2% strain. The area measured under the elastic portion of the stress-strain curve represents the resiliency of a material. The ultimate tensile strength is the material's ability to withstand load before fracture. The toughness of a material is the ability to absorb load and resist fracture. Dental ceramics are brittle in nature and cannot undergo measurable plastic deformation before fracture.

When a dental ceramic restoration contacts an opposing cusp, a tensile stress develops in the ceramic material. At a critical load, a crack initiates and propagation will occur. When a crack or internal flaw is present in the core, it will require a lower load and tensile strength to initiate the failure. The flexural strength test is one of the most well-established methods to evaluate dental ceramics. It is a method to measure material deformation behavior and strength. Flexure strength represents the greatest stress endured by the material before fracture. Flexural strength tests can be performed in uniaxial or biaxial loading arrangement with different setups such as: three-point flexural test, ball-on-ring and ring-on-ring (equibiaxial) flexural strength.

For ceramic materials, it has been suggested that the ring-on-ring test can provide the best information about the behavior of the material. The test subject's ceramic material is placed in a multi-axial tensile condition. It distributes the stress over a large area of the material and minimizes the stress formation at the edges of the test specimens.

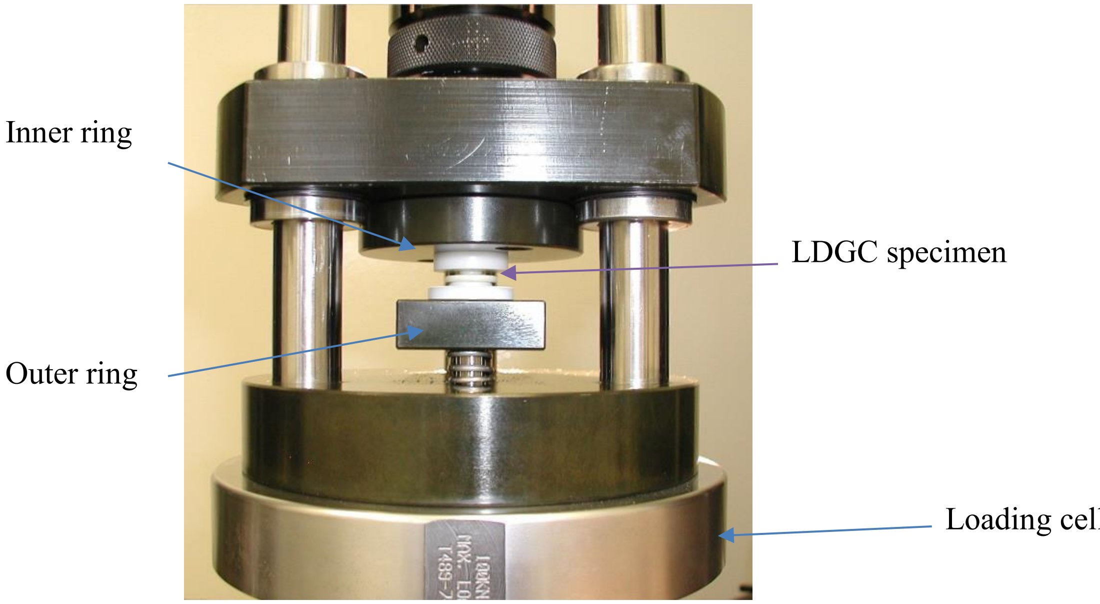

Figure 2 Equibiaxial flexural strength test.

Equibiaxial testing machine apparatus is composed of 5 major parts:

1. Load rod
2. Ball
3. Inner ring
4. Outer ring
5. Supporting platen

The test specimen’s specimen thickness should lie between

$$\frac{D_S}{10} \le h \le \sqrt{\sigma_f \, D_S^2 / E},$$

Where,

Ds: the supporting ring diameter

∂f: the expected equibiaxial fracture strength in units of MPa.

E: Modulus of elasticity in units of MPA

And for selecting the specimens and supporting ring diameter such as

D: the test specimen diameter in units of mm for the circular test specimens.

The ring/support ring ratio should lie between

$0.2 \leq DL/DS \leq 0.5$,

where DL is the diameter of the load ring and DS is the diameter of the support ring. The pair of rings used in this investigation included a 5.0 mm load ring and an 11.0 mm support ring.[51]

Monotonic load to failure test

Load to failure is an important test to predict how dental restoration may behave in the clinical situation. One of the most important variables that should be considered is the pressure contact area. Shrotriya et al 2003 conducted a laboratory study to investigate if the size of the indenter would affect the load required to initiate a subsurface radial crack in cemented ceramic restorations. It was found that a small spherical indenter does not create clinically relevant damage.[52] When a 20 mm diameter spherical indenter was used, a radial crack without a surface cone crack was achieved.[51] Researchers believe that to induce radial cracking without cone cracks, a large indenter must be used. In 2010, Kelly et al conducted a laboratory study to investigate a protocol for replicating the clinical failure of ceramic crowns.[33] The protocol should create a radial crack from the cementation layer with facture features similar to what has been reported clinically. No contact damage should be observed. The idea behind this protocol was to produce an ideal crack system by using a large spherical indenter. In that study, a loading piston with a 40 mm or greater radius was machined onto the loading piston to create a contact area in the range of 0.5 mm – 3.0 mm in diameter.[53] With a large spherical diameter loading piston, the contact pressure does not increase as fast as it does with small diameter loading pistons and hence it reduces the incidence of Hertzian contact cracks. The load recorded with 40 mm sphere was within the clinically relevant range. [53] When the spherical indenter radii is less than 40 mm, the load to failure required for a ceramic specimen was outside of the recorded chewing load range produced by humans.

After carefully reviewing of the literature there are no studies that have investigated the effect of adjustment size in the intaglio surface and its effect on strength. Moreover, there is no study that evaluated post adjustment healing protocols and their effect on the load to failure in a clinically relevant monotonic load to failure test.

Hypotheses:

There are 4 research hypotheses.

1. All specimens are in the divested condition. There will be no difference in the strength of diamond-adjusted and "repaired" e.max Press restorations compared to the non-adjusted e.max Press.

2. All specimens are in the divested condition. There will be no difference in the monotonic load to failure (contact pressure) of diamond-adjusted and "repaired" e.max Press restorations compared to the non-adjusted e.max Press.

3. All specimens are in the natural glaze condition. There will be no difference in the strength of diamond-adjusted and “repaired” e.max Press restorations compared to the non-adjusted e.max Press.

4. All specimens are in the natural glaze condition. There will be no difference in the monotonic load to failure (contact pressure) of diamond-adjusted and “repaired” e.max Press restorations compared to the non-adjusted e.max Press.

CHAPTER III

MATERIALS AND METHODS

The flexural strength of ceramics is probabilistic in nature and consequently enough specimens, generally greater than 20, must be tested to reduce the statistical uncertainty with its determination.[54]

Materials were composed of factors, IPS e.max Press that will be investigated with two tests. The methods measured the effect of diamond grinding of 0.4 mm (d) of e.max specimens with a thickness of 1.0 mm. An adjustment depth of 0.4 mm was selected as result of a pilot test during which it was observed that failure did not necessarily originate at the ground area when adjusted to shallower depths. Four adjustment depths (0.1, 0.2, 0.3, and 0.4) were tested in a ring-on-ring equibiaxial flexural strength test setup. Only at 0.4 mm depth of adjustment, did the fracture originate from the adjustment spot.

The following materials were tested:

Material (IPS-EP): IPS e.max Press ingot HA shade A1, disk-shaped specimens, 15 mm in diameter × 1 mm in height were prepared and fabricated according to the manufacturer's specifications and were subsequently modified as shown in Tables I and II. For the equibiaxial flexural strength test, each group consisted of 40 specimens. For the monotonic load to failure test, each group consisted of 20 specimens. Testing was performed in 2 parts, Part I and Part II. Part I specimens were divested and entered the test protocol, while Part II specimens were divested and then received a natural glaze according to the manufacturer's instructions prior to entering the test protocol.

Table 1 Biaxial flexural strength test groups (Part I).

Groups	**Treatment**
Control (G1)	Discs with no adjustment.
Adjustment (G2)	Discs were ground with a diamond bur on the cementation surface.
Acid Etch (G3)	Discs were ground with a diamond bur on the cementation surface and etched with hydrofluoric acid 9% for 20 sec.
Glaze (G4)	Discs were ground with a diamond bur on the cementation surface and were placed in the furnace for natural glazing.

Groups in this table entered the test directly after divesting

Table 2 Monotonic load to failure test groups (Part I).

Groups	**Treatment**
Control (G5)	Discs with no adjustment were cemented with (Multilink resin cement) to a supporting G10 epoxy resin block.
Acid etch (G6)	Discs were ground with diamond bur in the cementation surface. Discs were cemented with (multilink resin cement) to a supporting G10 epoxy resin block.
Glaze (G7)	Discs were ground with diamond bur in the cementation surface. Discs were placed in the furnace for natural glazing then were cemented with (multilink resin cement) to a supporting epoxy resin block.

Groups in this table entered the test directly after divesting

Table 3 Biaxial flexural strength test groups (Part II).

Groups	Treatment
Control (G8)	Discs with no adjustment.
Adjustment (G9)	Discs were ground with a diamond bur on the cementation surface.
Acid Etch (G10)	Discs were ground with a diamond bur on the cementation surface and etched with hydrofluoric acid 9% for 20 sec.
Glaze (G11)	Discs were ground with a diamond bur on the cementation surface and were placed in the furnace for natural glazing.

Groups in this table received natural glazing cycle after divesting then entered the test

Table 4 Monotonic load to failure test groups (Part II).

Groups	**Treatment**
Control (G12)	Discs with no adjustment were cemented with (Multilink resin cement) to a supporting G10 epoxy resin block.
Acid etch (G13)	Discs were ground with diamond bur in the cementation surface. Discs were cemented with (multilink resin cement) to a supporting G10 epoxy resin block.
Glaze (G14)	Discs were ground with diamond bur in the cementation surface. Discs were placed in the furnace for natural glazing then were cemented with (multilink resin cement) to a supporting G10 epoxy resin block.

Groups in this table received natural glazing cycle after divesting then entered the test

Wax pattern fabrication

A metal mold with an upper and lower member (Fig. 3) was used to form the wax pattern disc. The patterns were 15 mm in (d) × 1.0 mm (h). Patterns were produced from modeling wax made for use with a Bunsen burner (GEO Classic, Renfert Co. USA).[55] The wax has stress-free cooling properties, very low shrinkage and high accuracy and precision.[55] The wax was heated in a wax pot. Once the wax was completely molten, a stainless-steel measuring spoon was used to pick up and carry the wax into a Bunsen burner flame for 5-7 seconds. The molten wax was poured into the metal mold until the mold was completely filled. After pouring the specimens, the wax was allowed to cool for 2 minutes and excess wax was removed by scraping with a sharp blade. The wax patterns were separated and stored until the e.max Press specimens were fabricated. Wax patterns were sent to Apex dental laboratory (Madison, WI) for fabrication of the e.max Press specimens.

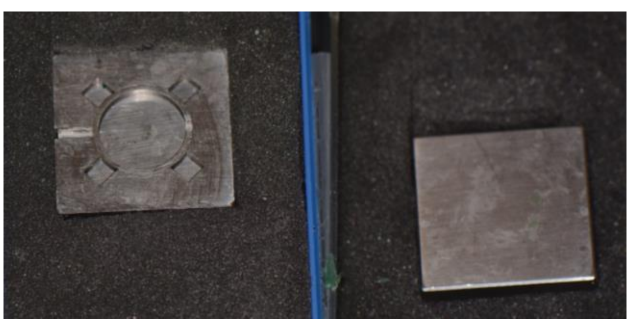

Figure 3 Metal mold to fabricate wax patterns.

Specimen selection

Wax patterns were inspected under 10× magnification (Fig. 4). Only specimens with a homogenous surface, free of voids and imperfections, were selected for inclusion in the study. Specimens were examined by two examiners.

Figure 4 Microscope evaluation.

IPS e.max Press specimen fabrication

Following the manufacturer recommendations, 8-gauge wax 5 mm long, was used to connect the wax patterns to the investment ring.[56] Pro-Art wax (Ivoclar Vivadent Inc) was used to seal the connection. Using a 200-g investing ring sprue guide, the patterns were oriented at 60 degrees and maintained at a distance of 10 mm from the silicone ring.[56]

Investing

A size 200 silicone ring gauge was carefully positioned so as not to damage the wax patterns. Following manufacture recommendations, 200 g of phosphate-bonded (IPS Press VEST Speed, Ivoclar Vivadent) with 32 ml of special liquid (IPS Press VEST Speed, Ivoclar Vivadent) and 22 ml of distilled water was mixed for 2.5 minutes in a vacuum mixer. The mixture was carefully poured into the silicone ring to the reference point and it was allowed to set for the recommended 45 minutes.

Preheating

After 45 minutes, the ring gauge and ring base were removed with a turning movement. The burnout oven was preheated to 850 C. The investment ring was placed in the preheated furnace (Vulcan Multi-Stage Programmable furnace, 3-130, 120V, Dentsply) toward the rear wall. The manufacture recommends that the ring is tipped and the opening is facing down.

Pressing

A cold (room temperature) IPS e.max Press ingot is inserted into the hot investment ring. The powder-coated (room temperature) Alox plunger is positioned into the hot investment ring. The completed investment ring is positioned at the center of the hot press furnace using investment tongs. The pressing program is selected according to the size of the investment ring and ingot to be used.

Divesting

After cooling to room temperature (60 minutes), the length of the Alox plunger was marked on the investment ring. Using a separating disc, the ring is cut at the mark and a plaster knife is used to break the ring. Rough divestment is performed using glass polishing beads at 4 bar pressure, then a subsequent fine divestment is carried out with glass polishing beads at 2 bar pressure. Ceramic residue on the Alox plunger is removed with alumina (100 microns). Invex liquid is used to remove the reactionary layer that develops on the ceramic specimens. Invex liquid contains ≤1 % of hydrofluoric acid.

Finishing

The sprues are cut off using a fine diamond disc. Any residual reactionary layer on the surface was removed with a fine diamond rotary cutting instrument.

Sample preparation

The specimens were randomly assigned to the groups. Each specimen was assigned a number which corresponded with the experimental groups. The specimens were saved in a case which is labelled with the specimen number. The thickness of each specimen was determined with a digital Micrometer (Mitutoyo IP65 series 342-27). Each specimen was measured at 3 different points around the center of the disc and then the mean was calculated. A 15 mm diameter circle with a center point was printed on transparent sticker paper. This template helped standardize the diamond rotary cutting tool adjustments made on the samples. The inner circle is 11 mm in diameter and was used for positioning the specimen in the equibiaxial loading apparatus.

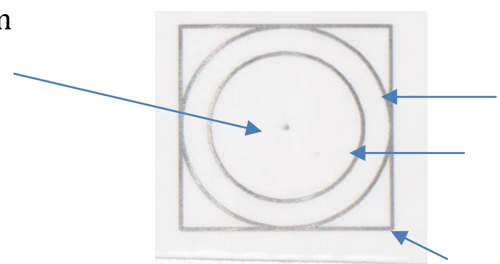

Figure 5 e.max discs. specification

All adjustments to the ceramic specimens were done with a milling machine (AF30 milling machine by NOUVAG) (Fig. 5). The ceramic specimens were positioned on the milling machine table using a custom-made positioner (Fig. 6). The positioner was made using low expansion stone (Resin rock, type IV stone), and a medium viscosity polyvinyl siloxane. The polyvinyl siloxane was located only at the periphery of the space holding the ceramic specimen.

The drilling handpiece was positioned perpendicular to the floor and the specimen positioner parallel with the floor. The adjustment depth (0.4 mm) was controlled by the milling machine micrometer. All adjustments were made at the marked center of the clear sticker using a dialite diamond rotary cutting instrument (856DEF.016, Brasseler, USA) and water. The adjustments were made at 10,000 rpm with light pressure.

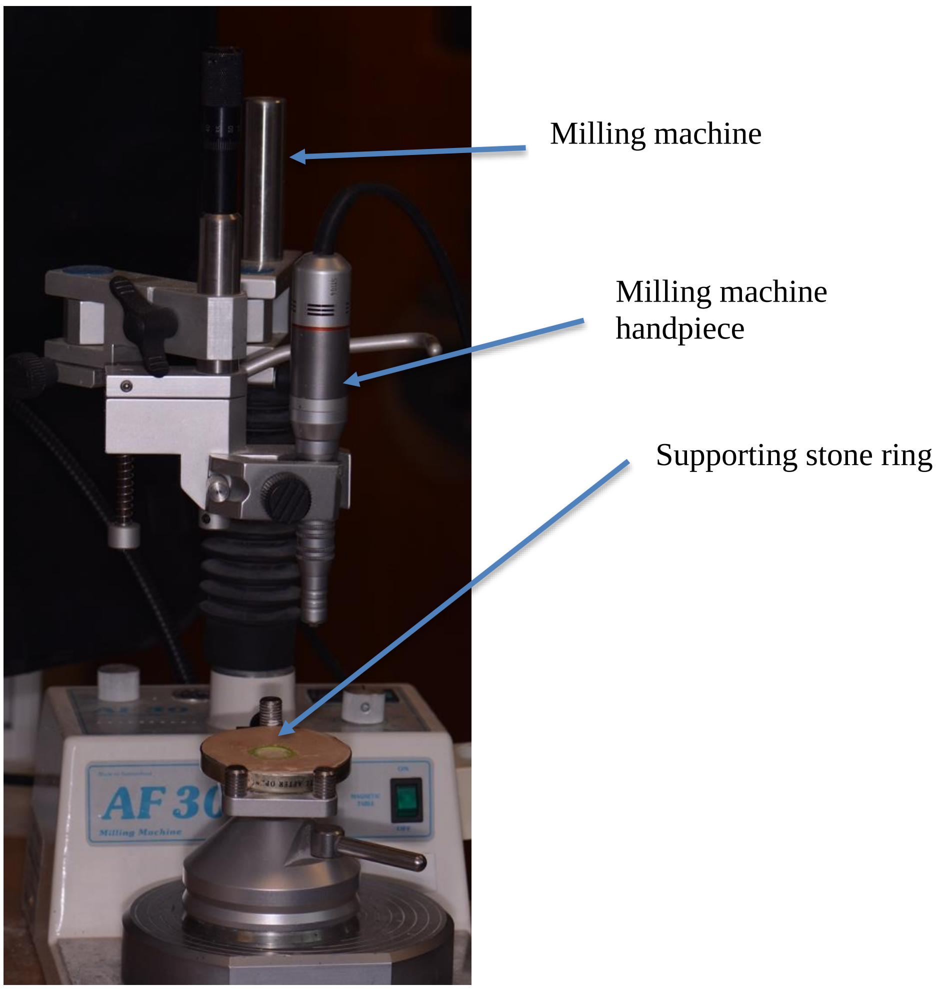

Figure 5. Milling machine

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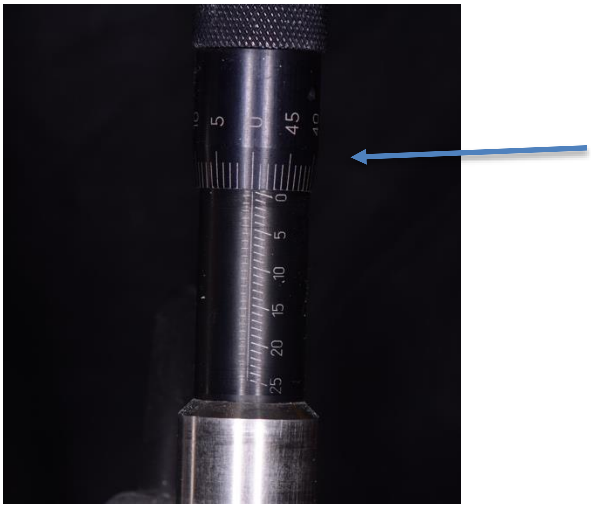

Figure 6. Gauge depth.

Step II in preparations

Acid etch treatment:

Group 3 were subjected to an acid etching treatment for 20 seconds on the adjusted area with 9.5% hydrofluoric acid gel (Bisco Inc., Schaumburg IL, USA), as recommended for clinical practice.

Glazing treatment:

Group 4 and 7 were placed in the furnace (Vita Vacumat 500, Zahnfabrik H.Rauter GmbH &Co.KG) for glazing treatment following the simulated clinical adjustments. The glazing protocol followed the manufacturer’s recommendations (Table 5).

Figure 7 Glazing furnace

Biaxial flexural strength

The specimens were centered on the supporting ring. The loaded surface of each specimen was covered with a clear sticker (0.05 mm) to distribute the load equally and to aid centering the disc in the testing apparatus. The diamond-adjusted side is facing down as it represents the intaglio surface. The specimens were loaded at 0.5 mm/min until failure and the failure load was recorded.

Monotonic load to failure

Sample preparation:

For the monotonic load to failure test, e.max Press discs were cemented to G10 epoxy resin blocks (G10). The epoxy possesses an elastic modulus similar to dentin. Prior to cementation, the cementation surface of the block was roughened with 25 micron aluminum oxide for 20 seconds, at a distance of 15 mm and pressure of 2.8 bar.

Groups 5, 6, and 7 will be tested in a monotonic load to failure test. Group 6 and 7 will be adjusted with a fine diamond rotary cutting instrument and a new diamond was used for every specimen. All specimens were adjusted at the same position using the sticker. Adjustments were made as previously described above.

Group 7 were placed in the furnace for natural glazing. The furnace was programmed according to the manufacturer recommendations for glazing. Protocol is illustrated in 5.

Table 5. Firing cycle used to produce a natural glaze.

IPS e.max	B	S	t↑	T	H	V_1	V_2
Glaze Firing	403C	6:00 min	60C	770 C	1:00 min	450 C	769 C

Group 6 specimens were cemented on the resin block according to the Multilink cementation protocol. The samples were treated with 5% hydrofluoric acid for 20 seconds then cleaned with water and dried. The etched surface was treated with Monobond and allowed to react for 60 seconds and then air dried. The cementing surface was scrubbed with a 1:1 mixture of self-etching primer for 30 seconds and air dried. Multilink cement then was dispensed onto the treated surface of the specimen and seated. A five-kilogram load was used placed on the specimens and light curing (Kerr) initiated the polymerization process, Fig 6. The same cementation protocol was applied for groups 5 and 7 as well.

The load to failure was calculated using the following relationship described by Lawn et al[57]:

P = contact pressure between the sphere indenter and the tested material surface.

$$P = (3E_1/4Kr)^{2/3} \cdot L^{1/3}/\pi$$

$$k = 9/16 \cdot [(1-v_1^2) + (1-v_2^2) \cdot E_1/E_2$$

E_1 = Elastic modulus of epoxy resin

E_2 = Elastic modulus of the spherical indenter material

v_1 and v_2 are the respective Poisson's ratios

L: Applied load

r: Spherical indenter radius

Mechanical testing:

Specimens were loaded with a 6.5 mm diameter piston with 50-mm radius on the loading point. Specimens were cushioned with a clear sticker of 0.05 mm thickness at a cross head speed of 0.5 mm/min. The loading cell was a 5-KN (Figure 9). Because peaks loads are not always visible on the load versus time plot or because loads oftentimes increase following failure, a measuring microphone is necessary. Acoustic events were recorded with a precision measuring microphone (Model M53; LinearX Systems, Inc, Tualatin, Ore). The microphone was used and positioned as shown in Figures 18 and 19. The microphone was situated very close to the specimen but did not contact it. Most humans can hear sound frequencies between 1-5 kHz. Fracture sound frequencies are often greater than 20 kHz. An amplitude-versus time graphs will be generated using noise analysis software (pcRTA, Version 2.30; LinearX Systems Inc). In the noise-analysis control panel, the pink noise generator will be selected, and an American National Standards Institute-A (ANSI-A) weighted filter will be used with the dynamic range fixed between –60 to 120 dBm. The noise analysis was started simultaneously with the monotonic load to failure test. The recording was used to detect crack sounds and assisted with determination of the failure load.

Figure 8. Instron machine.

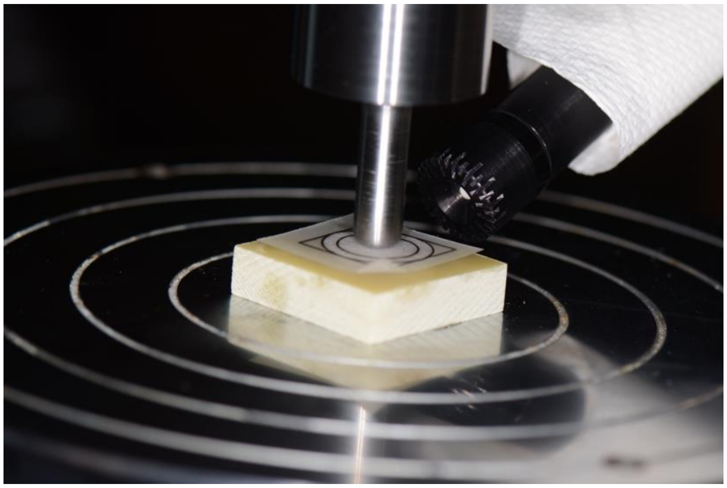

Figure 9. Monotonic load to failure setup.

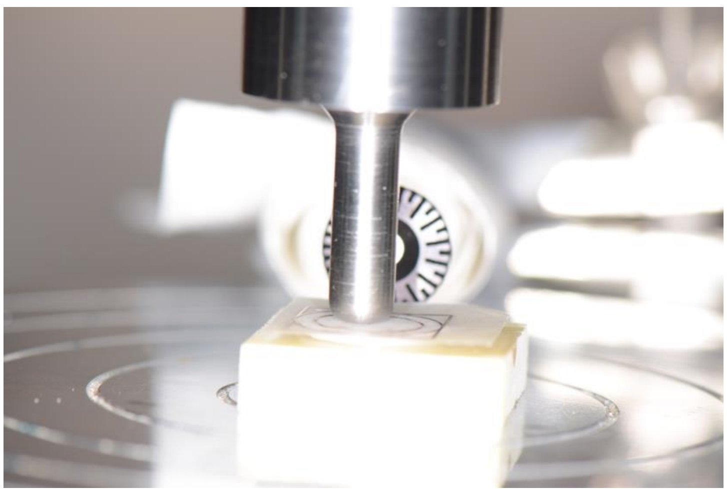

Figure 10. Monotonic load to failure setup close up.

Statistics:

Because brittle ceramic materials may contain flaws and defects as a consequence of production, failures are probabilistic in nature.[44,45] The strength of dental ceramics do not generally follow a normal distribution. Because each specimen will have different flaws the result is that strength will be different even when the mechanical test protocol is the same, the Weibull analysis is used. A previous study determined that using the two-parameter Weibull distribution with a maximum likelihood curve fitting is best practice for small data sets.[58]

The 2-parameterWeibull distribution is characterized by a shape (Weibull modulus) and a scaling (characteristic strength) parameter. They are estimated from facture data. When the Weibull of modulus is high it means, the data is tight together and the “standard deviation” is very low. On the other hand, when the Weibull modulus is low it means the data is spaced and the “standard deviation” is high. Materials with a low Weibull modulus will have a broad distribution of failure and will not exhibit the same reliability as a material with a high modulus.

A likelihood contour method was used for determining whether two Weibull distributions are statistically significantly different. This method is described in The New Weibull Handbook [Abernethy, RB. (2000). The new Weibull handbook. North Palm Beach, FL: Author]; however, simply stated, a horizontal slice is made in the 3-dimensional contour plot of the Weibull distributions being compared at equal likelihoods. The plot has the 95 % confidence bounds of the estimate for the Weibull shape parameter on the Y-axis and the 95 % confidence bounds for the estimate of the characteristic strength on the X-axis. If confidence bounds intersect, Weibull parameters are not statistically significantly different.

CHAPTER IV
RESULTS

Part I

Equibiaxial flexural strength

Regarding the equibiaxial failure group, Part I, a 2-Parameter Weibull Plot and likelihood ratio contour plot revealed a significant difference between the characteristic strength of the control group and the other groups. The control group ranked the strongest and the acid etched treatment ranked the weakest. There was a significant difference between G4 and G2. There is no significant difference between G3 and G4. G1 possessed the highest Weibull modulus while the lowest Weibull modulus was observed with G4 (Figures 11 and 12). The flexural strength of each group is given in Table 6.

Table 6. Equibiaxial failure strength, Part I.

Group	Flexural strength (MPa)
G1	187.6
G2	161.7
G3	160.2
G4	175.6

G1: control, G2: diamond adjustment, G3: Adjustment plus acid etch, G4: Adjustment and glaze

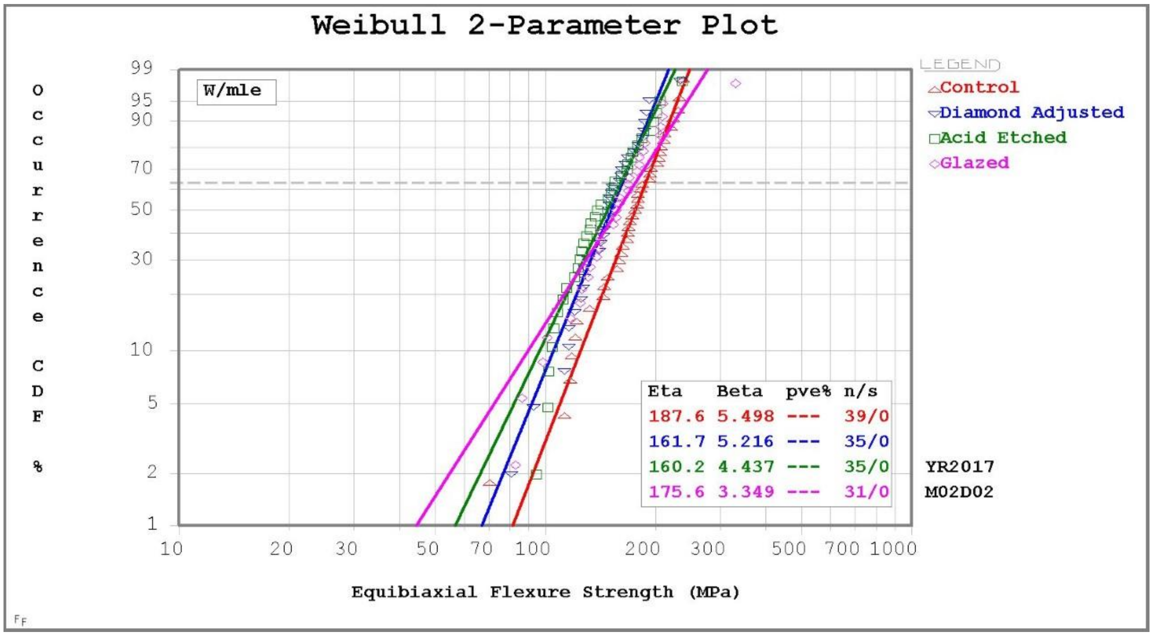

Figure 11. Two-parameter Weibull plot equibiaxial flexural strength part I.

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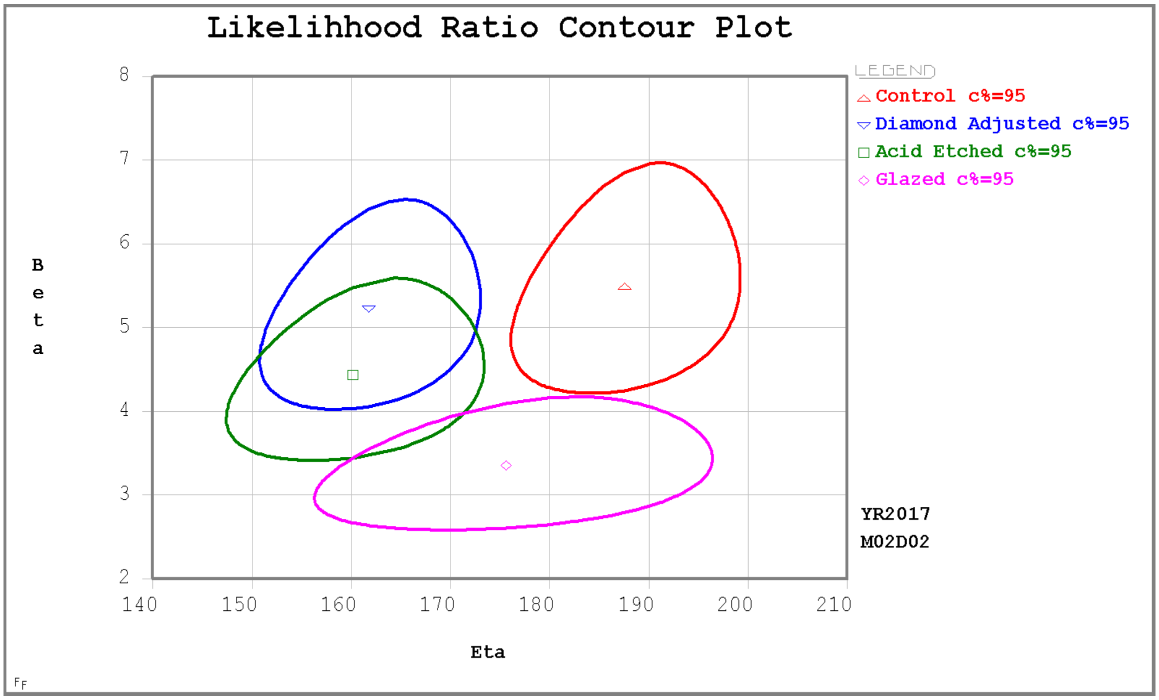

Figure 12. Likelihood Ratio contour plot equibiaxial flexural strength part I.

Monotonic load to failure:

Monotonic load to failure data is presented in Table 7. Regarding the monotonic load to failure group, Part I, the 2-Parameter Weibull Plot and likelihood ratio contour plot revealed no significant difference between the control and glazed groups. The diamond adjusted group was significantly different from the control group and the glazed group (Figures 13 and 14).

Table 7. Monotonic load to failure, Part I.

Group	Load to failure(MPa)
G5	122
G6	156
G7	94.52

G5: control, G6: Glazed and G7: Etched

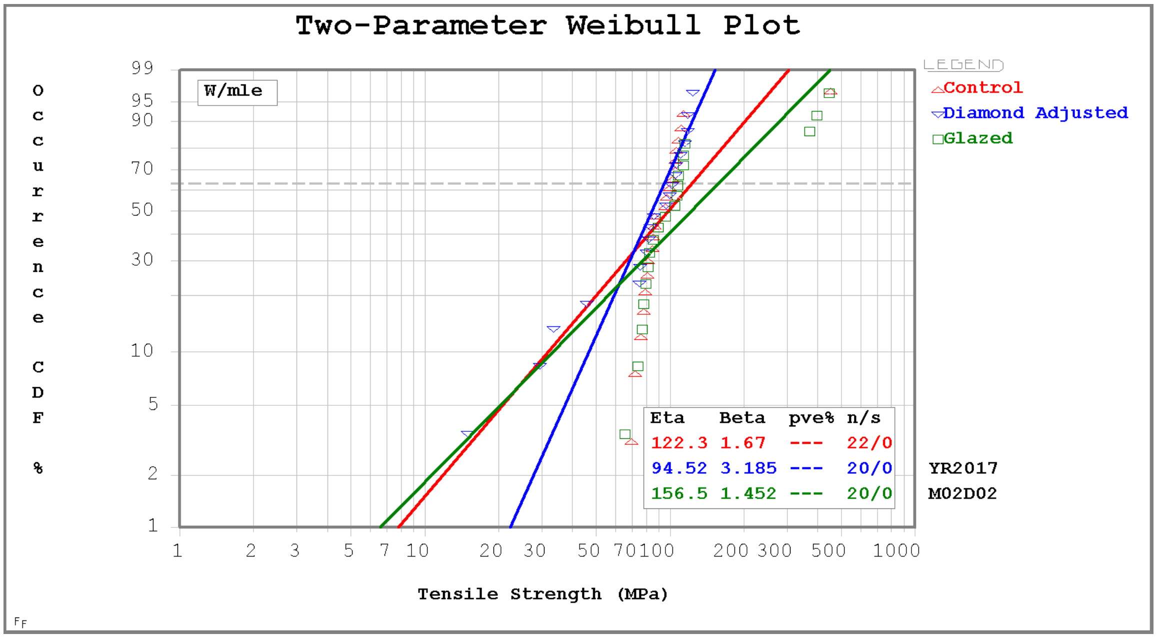

Figure 13. Two-parameter Weibull plot Monotonic load to failure part I.

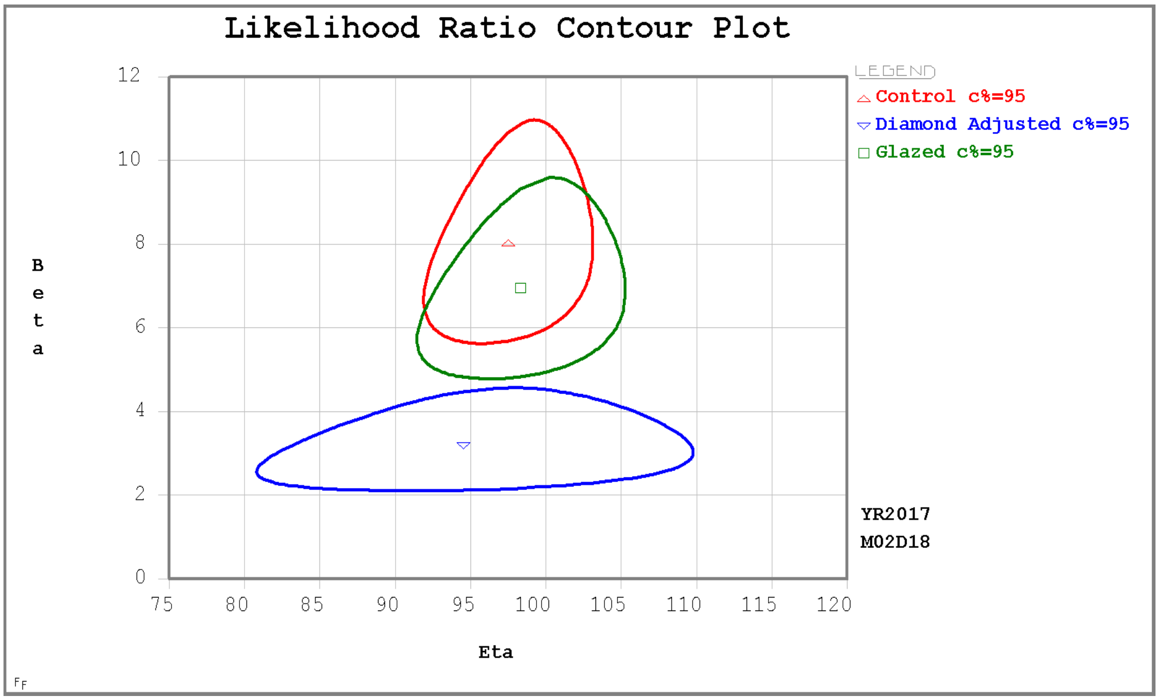

Figure 14. Likelihood Ratio contour plot Monotonic load to failure part I.

Part II

Equibiaxial flexural strength

G8 was the strongest while G9 ranked the weakest (Table 8). Regarding characteristic strength, a 2-Parameter Weibull Plot and likelihood ratio contour plot revealed a significant difference between the control group and the other groups. (Figures 15 and 16). There was a significant difference between G2 and G4. There was a significant difference between G9 and G11. There was no significant difference between G9 and G10. G8 possessed the highest Weibull modulus and the lowest Weibull modulus belonged to G11. All groups in Part II of the study demonstrated a higher strength compared to groups in Part I. Similarly, the Weibull modulus increased in all the groups except G9.

Table 8. Flexural strength results Part II

Group	Flexural strength (MPa)
G8	239
G9	168
G10	191
G11	203

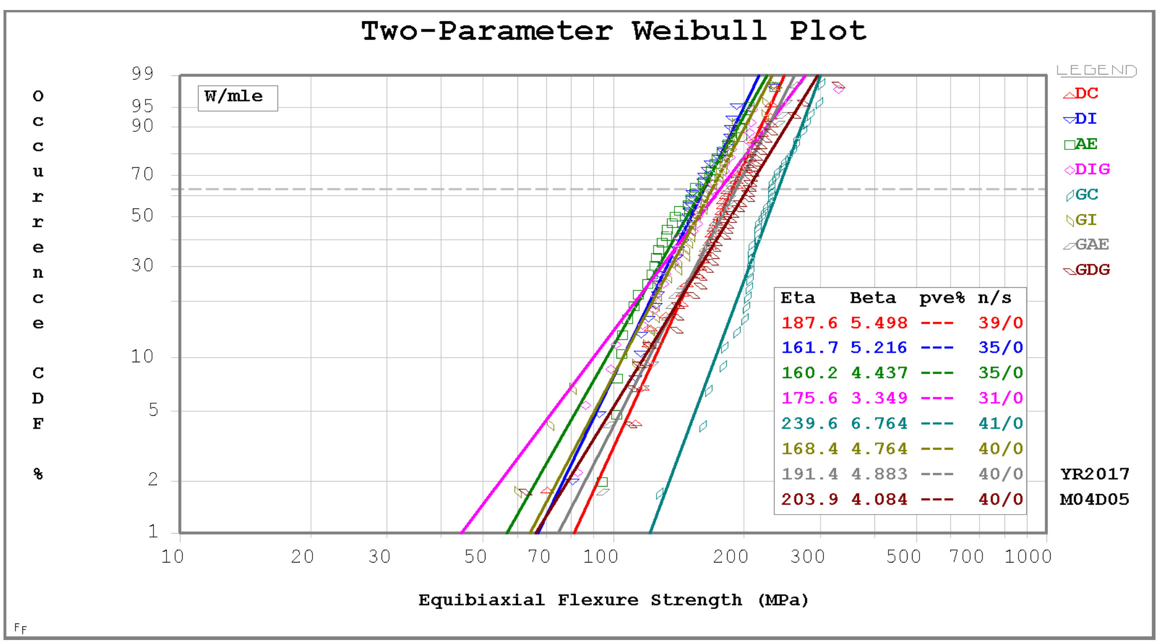

Figure 15. Two-parameter Weibull plot equibiaxial flexural strength Part II

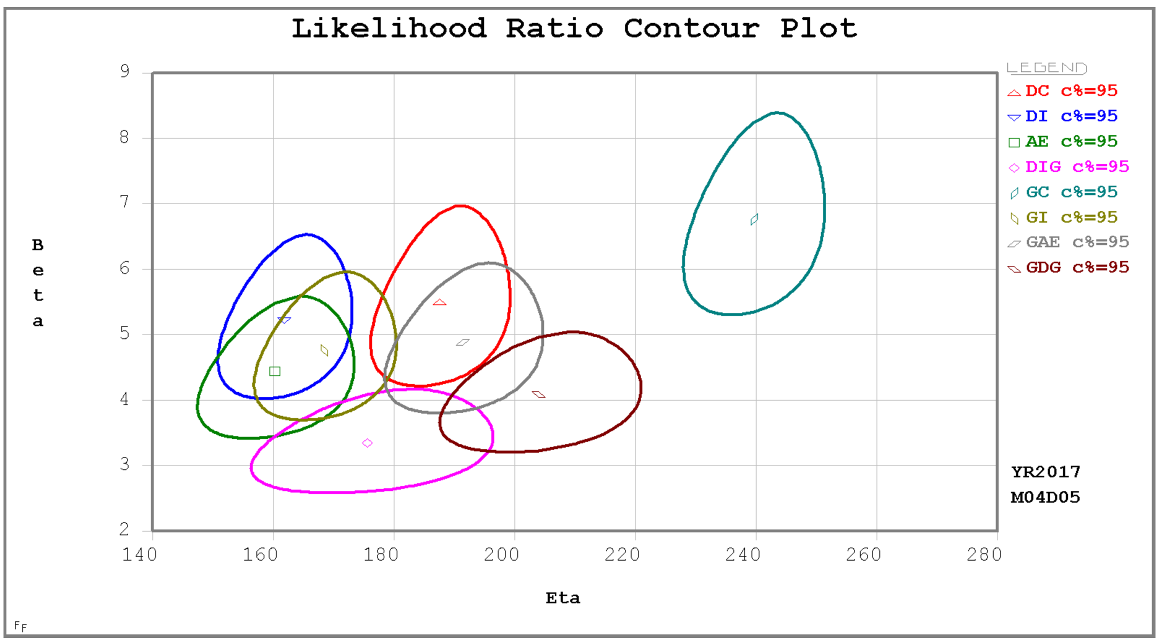

Figure 16. Likelihood Ratio contour plot equibiaxial flexural strength Part II

Monotonic load to failure:

Regarding monotonic load to failure (Table 9), and the 2-Parameter Weibull Plot and likelihood ratio contour plot (Figures 17 and 18) it was found that no significant difference existed between G12 and G13. There was a significant difference between G12 and G14. The control group in Part II exhibited a higher Weibull modulus compared to the control in Part I. G13 has a slight decrease in the Weibull modulus. The contact pressure of all the groups in Part II increased compared to the groups in Part I.

Table 9. Monotonic load to failure, Part II.

Group	**Load to failure (MPa)**
G12	123
G13	124
G14	115

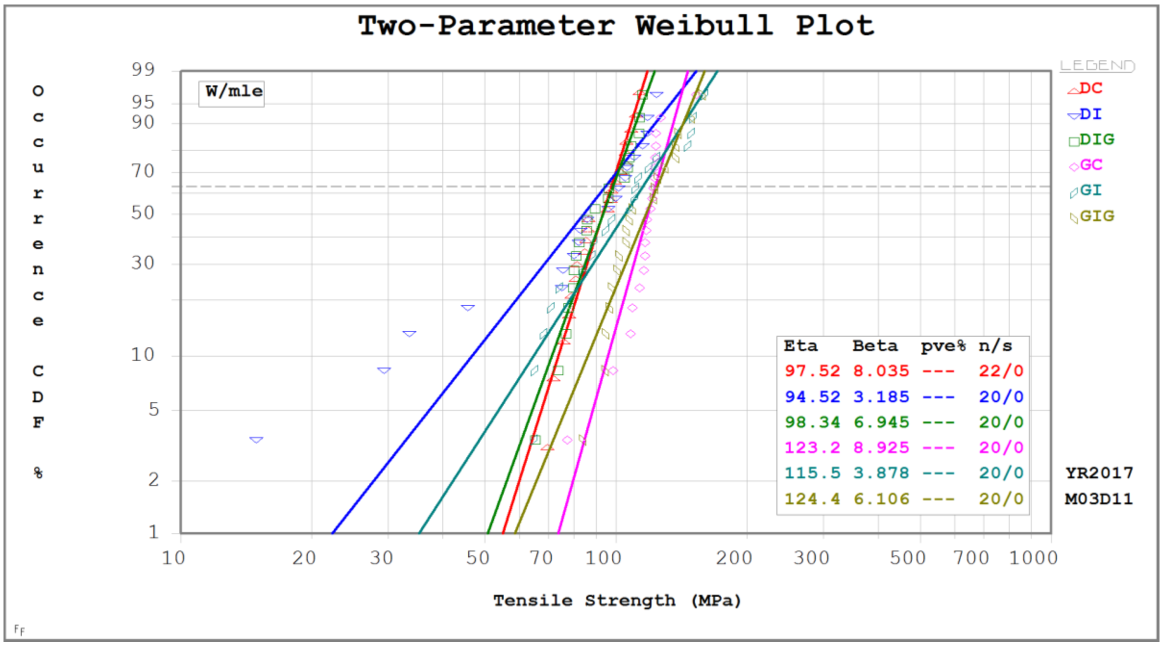

Figure 17. Two-parameter Weibull plot Monotonic load to failure Part II.

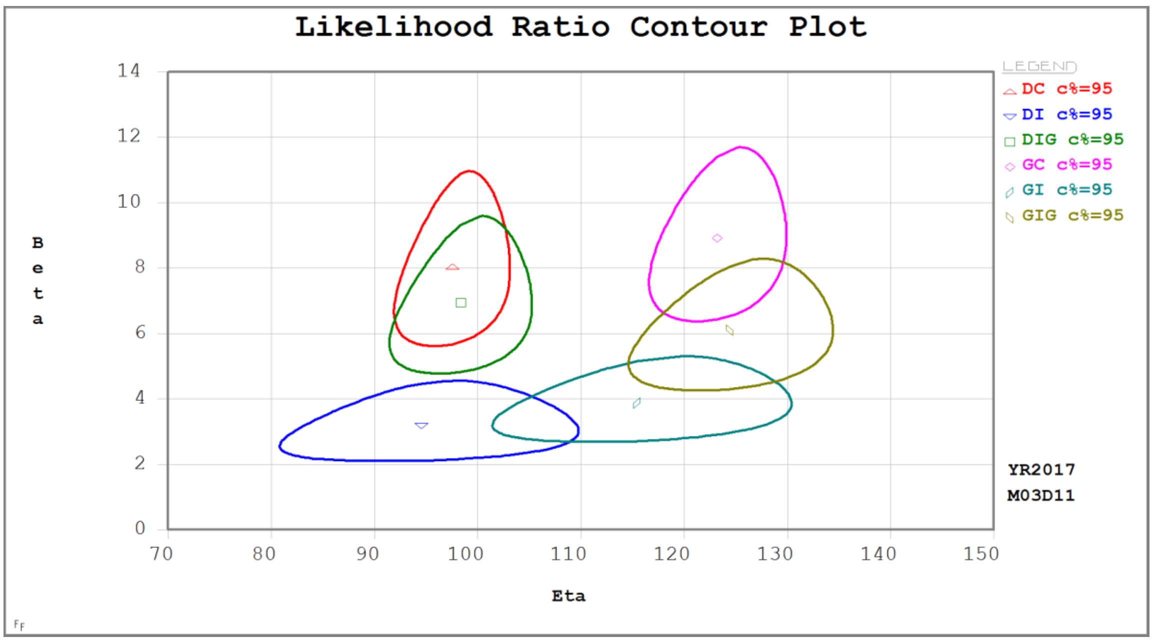

Figure 18. Likelihood Ratio contour plot Monotonic load to failure Part II.

CHAPTER V

DISCUSSION

The first null hypothesis was rejected as there was a significant difference between the control and damaged and repaired e.max Press specimens. The second null hypothesis was not rejected as there was no statistical difference between the contact pressure of the control group and the damage and repaired groups. The third null hypothesis was rejected as there was a significant difference between the control specimens and the damaged and repaired specimens. The fourth null hypothesis was not rejected as there was no statistical difference between the control group and the damaged and repaired groups.

Hung 2008, published a study on the effects of simulated clinical grinding and subsequent heat treatment on micro crack healing of a lithium disilicate ceramic. The result of the study was that grinding of lithium disilicate ceramics with diamond rotary cutting tools may introduce flaws and cracks, and therefore, subsequent heat treatments, veneer firing, or glazing are suggested. One of the limitations to this study is that the depth of the adjustment was not provided and the adjustments were performed on the "occlusal surface". Moreover, a ring-on-three-balls loading arrangement was used, which may lead to edge chipping from contact stresses. In the present study, a ring-on-ring test was used because, (1) it produces an equibiaxial stress state, and (2) since load is distributed over a larger area of the specimen, failures from contact stresses are minimized.[58]

The result of the current study showed that a glazing treatment improves the strength and load to failure of the material in general and damaged specimens in particular. In the biaxial flexural strength Part I of the study, when the specimens entered directly into the study protocol after divesting; after diamond adjustment the glazing helped increase the strength of the material (see table IV). Moreover, it was found that in the load to failure test Part I that glazing treatment resulted in damaged and repaired groups that were not significantly different from the control.

In the monotonic load to failure (contact pressure) test Part II of the study, all the specimens went through a natural glazing cycle before starting the experimental procedures. Two important findings were revealed. First, the Weibull modulus increased in general. It is believed that the glazing treatment healed the investing and divesting damage and as a result the data became more consistent. Secondly, the strength and failure load of the materials Part II of the study increased significantly compared to Part I of the study. This may mean that manufacturing defects have a significant impact on the strength of e.max Press lithium disilicate material. Moreover, glazing after divesting improved physical properties

The Weibull modulus describes the reliability of a material. The higher the Weibull modulus the more reliable the material is. Both the control groups and the groups that received a glazing treatment post-adjustment demonstrated a higher Weibull modulus compared with the divested or non-glazed specimens. The control groups exhibited the highest Weibull modulus. The diamond adjusted group from both parts of the study showed a low Weibull modulus. It appears that diamond adjustment to e.max Press lithium disilicate, with no further glazing treatment, can lead to material faults and defects that reduce the reliability of the material.

In the biaxial test, in both parts of the study, the level of reliability was reduced significantly after diamond adjustment. However, when the discs were cemented onto the G10 epoxy resin, post adjustment glazing showed an acceptable level of reliability with a Weibull modulus of 6. Malament et al 2001 reported that resin cement bonding increased the survival rate of Dicor restorations, and similarly the result of this study found that the resin bonded e.max specimens exhibit a strength similar to the control. Another observation is that the load to failure test revealed a lower contact pressure for all test groups. It is believed that the monotonic load to failure test placed more of the specimen volume under tension and compression, hence the lower loads to failure compared to the equibiaxial flexural test.

There are a few limitations of the study. First, it is a laboratory study. The results of the study are related to the specific material e.max Press (Ivoclar Vivadent) and Multilink resin cement. The study did not simulate oral fatigue condition; mechanical (cyclic loading), or chemical and thermal changes. Each of which may affect the performance of the e.max Press bonding in the long term. Finally, the specimen geometry is different than a normal dental restoration.

CHAPTER VI

SUMMARY AND CONCLUSIONS

- A glazing treatment improved the physical properties of adjusted IPS e.max Press discs when subjected to biaxial flexural test and monotonic load to failure.
- Diamond adjustment to lithium disilicate reduced the reliability of the material. When clinical adjustments are made on the IPS e.max Press intaglio surface, a subsequent glazing treatment is recommended.
- The strength of the material following glazing was similar to the control.
- The average load to fracture of the cemented discs was within the recorded range of human biting forces.
- A majority of the cracks started from the intaglio surface by means of radial cracks and without evidence of surface damage.
- The groups followed a similar rank order in terms of strength; the control ranked the strongest while acid etch ranked the weakest.
- The testing methodology appeared to replicate clinical failure loads.

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