Submitted by Seth Dressman Caldon in partial fulfillment of the requirements for the degree of Master of Science in Oral Biology.

Accepted on behalf of the Faculty of the Graduate School by the thesis committee:

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MICROTENSILE BOND STRENGTH COMPARED BETWEEN CAD/CAM FELDSPATHIC AND RESIN NANO CERAMICS

BY

Seth Dressman Caldon

A THESIS

Submitted in partial fulfillment of the requirements
For the degree of Master of Science in the
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in the Graduate School of
The Uniformed Services University of the Health Sciences

FORT BRAGG, NORTH CAROLINA
2015

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LIST OF ABBREVIATIONS

CAD/CAM- Computer Aided Design/Computer Aided Milling

CEREC- Chairside Economical restorative Esthetic Crown

SEM- Scanning Electron Microscopy

σ_f- Fracture strength

K_{IC}- Fracture toughness

c- Flaw size

Y- Geometric constant

MPa- Mega Pascal

GPa- Giga Pascal

s- Bond Strength

L- Test Load

A- Adhesive Area

μTBS- Microtensile Bond Strength

'A'- Adhesive Failure

'B'- Cohesive Failure in Ceramic

'C'- Cohesive Failure in Cement

'D'- Mixed 'A' and 'B'

'E'- Mixed 'A' and 'C'

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ABSTRACT

Objectives: To investigate the microtensile bond strength (μTBS) and failure mode of resin cement bonded to feldspathic and resin nano ceramic CAD/CAM crowns.

Methods: Feldspathic and nano resin ceramic blocks, which are designed for the CEREC CAD/CAM system (Sirona, Charlotte, NC), are to be used for the study. Four CEREC Vitablocs Mark II (fine-particle feldspar ceramic blocks) I14, size 12x14x18 mm^3, (Vita, Bad Sackingen, Germany), and four 3M Lava Ultimate (resin nano ceramic) size 14L (3M ESPE Saint Paul, MN) were used. Block surfaces were treated with hydrofluoric acid or air abrasion before application of a layer of Nexus 3 resin cement. Z100 composite was then incrementally cured to the cement in 2 mm increments to a thickness of 5 mm. Blocks were cut creating 50-60, 1 mm^2 slabs. The slabs were stored in distilled water at 37°C for 24 hours. 12 slabs were chosen at random from each block. Tensile stress was applied to slabs with an Instron Universal machine until failure. 30X light microscope was used to confirm adhesive failure, and the force was recorded in Mpa. The measured MPa were then compared statistically with a Two-Factor ANOVA and Tukey test

Results: Microtensile strength was not statistically different within the blocks A-D for both groups. There was a significantly different between Vitablocs Mark II and Lava Ultimate with 18.5Mpa (+/-3.7) and 47.6MPa (12.2+/-) respectively.

Conclusion: Within the limitations of this study, Lava Ultimate has a significantly higher bond strength compared to Vitablocs Mark II.

INTRODUCTION

To stand up to long term wear, a restoration needs to be hard and flexible. Unfortunately, these are antagonistic features. Hard structures tend to be resistant to wear but tend to be brittle and prone to fracture, while flexible materials are less resistant to wear. Teeth have evolved with hard enamel that is brittle, but is supported by flexible dentin. There is a continuing search for a material that provides both the hardness and flexibility to give long lasting results that are esthetically appealing as to mimic natural teeth. Lava Ultimate is a recently introduced product marketed by its manufacturers to provide the necessary esthetics, flexibility, and hardness. The development of chair-side Computer Aided Design/ Computer Aided Milling (CAD/CAM) has allowed providers to give this restoration to a patient in one relatively short appointment. The purpose of this study was to compare the use of CAD/CAM all-ceramics and how Lava Ultimate overcomes the faults, specifically fracture, of previous ceramic restorations by incorporating nano ceramics in a resin matrix.

Computer Aided design/Computer Aided Milling

First, it is important to briefly review the design and milling systems. Computer Aided Design/ Computer Aided Milling (CAD/CAM) systems are becoming more popular these days in the use of prosthodontics. Chairside milling provides a convenient and relative shorter dental treatment compared to the traditional laboratory procedure. The CEREC system (Sirona Dental

Systems) has dominated the market since its arrival over 20 years ago (1) (2), and therefore is primarily discussed in this review as opposed to other systems such as E4D (Planmeca). The system was originally designed for inlays and onlays, which is the reason why most CAD/CAM research has been published on inlays (3). The newest of systems is the CEREC 3, which is capable of fabricating inlays, onlays, posterior crowns, anterior crowns, veneers, and fixed dental prostheses (FDP) frameworks (3). Fasbinder decribes the main concerns with CAD/CAM all-ceramics are postoperative sensitivity, color matching, marginal adaptation, and fractures (4). These concerns are briefly reviewed below with emphasis placed on catastrophic fractures.

Early studies on the CEREC system involved high levels of postoperative sensitivity. Sjorgen, Fasbinder, and Otto published results between 1991 and 2002 showing 9% to 13% of cemented CAD/CAM inlays with immediate postoperative sensitivity. Most of the cases resolved at the one month mark, but some lasted up to 7 months. All the cases resolved after the 7 months. Due to improvements of adhesive and luting techniques, more recent clinical studies have reported less postoperative sensitivity (5) (6) (7). Molin et al, 2000, and Heymann et al, 1996, found no sensitivity at recall appointments within their clinical studies (6). Fasbinder, in 2005, published one of 80 CAD/CAM inlays cemented had postop sensitivity at the one week recall. However, no sensitivity was reported in the second week or for the remainder of the 3 year study (6).

Most CAD/CAM ceramic blocks are of a single color, or monochromatic. Getting a color match with monochromatic blocks is very difficult. Most of the color comes from the milled block and the luting cement (8). Molin and Karlsson compared IPS Empress (Ivoclar Vivadent), Mirage (Myron International) and Vitablocs Mark II (Vita Zahnfabrik) to a cast gold inlay in 20 patients. All four restorations were placed in each patient and were followed for five years. Empress changed from a 15% baseline color mismatch to 30%, the Vitablocs Mark I changed from 15% to 40%, and the Mirage changed from a 25% to 50% mismatch (9). Fasbinder published a study in 2001 that showed the Vitablocs Mark II 16% baseline mismatch increasing to 46% after three years, but indicated that color shift was due to the teeth rather than the color of the restoration (4). Therefore, all-ceramic restorations have shown to provide a lasting color match.

The accuracy of the milling process is important for fabricating a restoration that fits intimately. Poor marginal adaptation is significant because it can lead to cement dissolution, micro leakage, increased plaque retention, and secondary caries (10) (11). Sadowsky stated that marginal adaptation has not been well documented over the long term, even though marginal discrepancies occur in 40% of restorations after three years, and 74% after 10 years. He follows with the marginal discrepancy being attributed to wear of the resin cement, but points out that the marginal discrepancy has not been associated to caries in the long-term studies (12). Mclean suggested that 120 microns should be the limit for a clinically acceptable marginal discrepancy (13). Christenson reported the clinically detectable range for subgingival margins was between 34 and 119 microns, and supragingival margins between 2 and 51 microns (14). The limitations of the CAD/CAM system are related to the optical impression and the use of the milling burs. Reisch stated that there are two phenomenons that occur when making an optical impression known as 'rounded edges' and 'overshooters'. The 'rounded edges' occur from the finite scanning resolution of the measuring system that makes the digital impression appear slightly rounded. 'Overshooters' are virtual peaks near the edges created by the software that are not present clincially (15). Also, the diameter of the milling bur that cuts the intaglio surface of the restoration may be larger than some parts of the tooth (15). Asavapanumas, also added that increasing the degree of curvature in the margin increases the marginal discrepancies in milling (16). In general, studies have demonstrated that internal gap widths are larger than marginal gaps, and most discrepancies are within the clinically acceptable range (15). Bindl compared slip cast (In- Ceram Zirconia), Heat-pressing (Empress II), and CAD/ CAM crown copings (CEREC inLab, DCS, Decim and Procera). He measured the marginal gaps with SEM. The marginal gap of the slip cast was 25(+/- 18) microns, which was significantly smaller than the Empress II 44(+/-23) microns. The Procera had a marginal gap of 17 (+/- 16) microns and Decim had a marginal gap of 23 (+/- 17) microns. The Cerec inLab marginal gap was 43 (+/- 23) microns (17). The DCS had a marginal gap of 33 (+/- 20) microns. All the crowns had similar internal fit (17). This information shows that the marginal and internal fit of the

CAD/CAM is comparable with the conventional methods of all-ceramic crowns, and are well within the clinically acceptable fit of 120 microns.

All the qualities discussed above demonstrate that the CAD/CAM crowns are reliable and dependable restorative alternative to the conventional metal ceramic crowns and direct restorations. A review by Hickel and colleagues, in 2001, evaluated annual failure rates of restorations in posterior stress bearing areas. Amalgam restorations had a failure rate of 0 – 7%, direct composites had a failure rate of 0 – 9%, glass ionomer had a failure rate of 1.4 – 14.4%, composite inlays had a failure rate of 0 – 11.8%, ceramic inlays had a failure rate of 0 – 7.5%, gold inlays had a failure rate of 0-5.9%, and the CAD/CAM ceramic inlays had a failure rate of 0 – 4.4%. They reported the majority of the failures were due to recurrent caries for the direct composite, amalgam, and glass ionomer restorations. Bulk fracture of the restorations or tooth caused the majority of the failures for the indirect restorations (12). Sjorgen and colleagues produced three reports on 66 Vitablocs Mark II over 10 years and found a survivability of 89.0% using the Kaplan – Meier method (5) (18) (4). While 89% is a decent success rate, there is still room for improvement to make the restorations a better long-term treatment option.

Survival of All-Ceramic materials

Comparing the success rates of all-ceramic crowns is challenging due to heterogeneity between studies (19). One major complication resulting in crown failure is fracture (12) (20) (21). Mormann et al, in 1991, published the results of the first 94 Vitablocs Mark I inlay restorations placed between 1985 and 1987. After following the cases for three years they reported only two fractures (22). Posselt and Kerschbaum, in 2003, placed 2,328 inlays and onlays in 794 patients and reported 35 failures from fractures over nine years (23). Otto and Denisco reported an 8 percent failure rate from fracture for 200 Vitablocs after a 10 year follow-up (7). The resistance to fracture of an all-ceramic crown is dependent on the core-veneered bond strength (when used), crown thickness, design of the restoration, and luting cements used to bond to the tooth (19). There are two types of notable ceramic fractures: the Hertzian cone crack (24), often resulting from surface damage on the occlusal surface that extends deeper into the restoration, and radial cracks that form at the cementation zone (25).

Fracture mechanics and fractography are both important fields utilized in assessing the cracks to help determine the reason for failure. Fracture mechanics associated with ceramic crowns was pioneered by the work of Griffith, Orowan, and Irwin, and is described by Kelly as using mathematical modeling to calculate energy and material strength to describe the reason of ceramic failure associated with flaws within the ceramic material (26). Griffith created an equation, $\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$, which relates applied stress and crack length at fracture, where σ_f is the fracture strength, K_{IC} is fracture toughness, c is flaw size, and Y is the geometric constant. This equation theoretically explains why microtensile bond tests show a significantly higher strength compared to macrotensile bond tests, which will be discussed in depth later (26).

Fractography is the study of the surface features and how they relate to crack propagation. Cracks go through a series of characteristics that change as the crack gains energy and velocity. Cracks begin as a smooth region termed the mirror, and then as energy increases secondary cracks may propagate giving a misty look, followed by the third region, hackle, where the secondary cracks are visualized branching in individual distinct paths (26). Quantitative fractography can be used to determine the source of the crack and calculate the stress at failure.

With the use of fractographic analyses and fracture mechanics, clinical fractures have been found to be a result of the radial crack formation that forms at major flaws in the ceramic (27). Thompson, in a study of fractured Dicor and Cerestore crowns, concluded that the fracture initiation is controlled primarily by the location and size of the critical flaw, and not by the specimen thickness (28).

With the use of CAD/CAM materials, inherent flaws from fabricating the material have significantly decreased because of standardized industrial processing (29) (30) (8), although flaws may still occur from the milling process. The radial cracks form from the occlusal load deforming the restorative material which then puts tensile stresses at the cement interface (26) (31).

Therefore, the majority of all-ceramic crown fracture research involves the study of the bonding interface strength along with inherent properties of the material such as flexural strength and flexural modulus (25). The ideal ceramic material would require zero flaws, have a high flexural strength, low flexural modulus, and adequate bond to tooth.

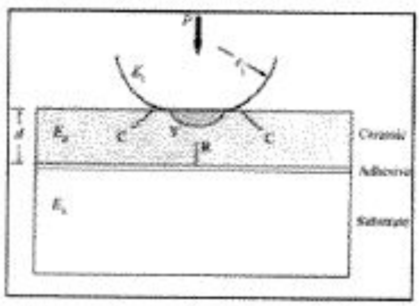

Figure 1. Schematic of brittle layer of thickness d and modulus E_C on a compliant substrate of modulus E_S, depicting sphere indenter of radius r_i. (Specimen radius r_c in general relation Eq. 1a is effectively infinite in the flat layer structure depicted here.) Damage modes: surface cone cracks (C); *quasi*-plastic yield zone (Y); inner-surface flexural radial cracks (R).

Figure from Lawn et al depicting cone and radial fractures

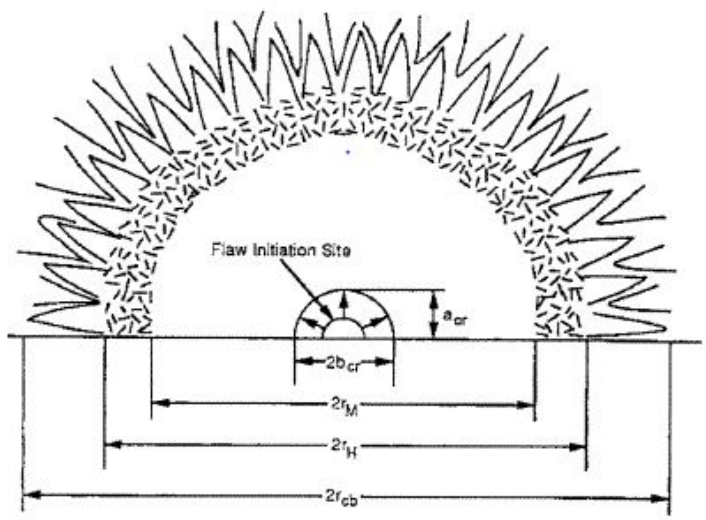

Figure 1. Schematic illustration of the fracture surface of a brittle material. Inner semi-ellipse represents the initial flaw, and outer semi-ellipse represents the critical flaw size. The subscripts M, H, and cb represent mist, hackle, and crack branching, respectively.

Figure from Thompson et al depicting fractography characteristics

Adhesive Cements

Clinical data strongly supports using adhesive cements and bonding the ceramics to teeth to achieve higher success rates (32). Luhrs states that adhesive cementation of ceramics requires a composite resin (33). Resin cements are hydrophobic. Other adhesive cements that are hydrophilic, such as compomers, will swell and induce fractures (33). It has been shown in several studies that the use of adhesive cements increases the fracture resistance of ceramic restorations (3) (34) (35). In Burke's review, one study, through photoelastic examination of load transformation mechanisms, showed that enhanced clinical performance of bonded ceramics is achieved by the transfer of stress through the tooth-crown interface (36).

The resin cements can be classified as active and passive (33). The active resin cements bond with the dentin hybrid layer. The passive resin cements bond via interlocking mechanical retention between the rough surfaces of the crown and prepped tooth. The uses of the passive cements have become less used because the active resins have superior characteristics (33). Ceramic surfaces can be roughened via etching with 4-10% hydrofluoric acid or sandblasting with 50 microns alumina oxide. Guarda et al, in 2013, performed fatigue and microtensile tests on IPS e.max Press ceramics (Ivoclar Vivadent) with a resin cement, and demonstrated that the hydrofluoric acid etching significantly increases the bond strength (37). When the ceramic restorations are silanated, the active cement can bind chemically to them (38).

The active resin cements can be broken down in to three categories: chemically cured, light cured, and dual cured. The chemically cured active resin cement begins the curing when it is mixed and has a certain working time. Light cure resin cements are activated by a light source so there is more control of the working time, and the dual cure resin cement is a combination of both light and chemical cured (38). The active resin cements require a procedure similar to placing posterior direct composites. The tooth needs to be etched, and the dentin primed, before the bonding agent is applied (33). There are many products that combine one or all the steps to reduce time and make a more user friendly product. Resin self-adhesive cements differ in that they don't require etch, prime, and bonding steps (38). Proos discussed the influence that the luting cement has on crowns. In the article, using finite element analysis, he compared an adhesive resin (active resin cement) and zinc phosphate. The study found that zinc phosphate had greater potential to fracture the ceramic, and adhesive resin had a stronger potential to transfer the load stresses to the tooth (39).

Testing Shear and Tensile Bond Strength

Considering the association of the ceramic fractures and adhesion interface, bond strength measurements are one of the main aspects to help identify durability of ceramic materials under load. Bond tests measure the stress needed to initiate fracture from the largest flaw (40). Bond test results are often separated into adhesive failure, cohesive failure, or mixed. Cohesive and mixed failures are often discouraged results because they reside mostly in the material and are not related to the adhesion interface (41). Bond tests available comprised initially of macro forms involving areas larger than 3mm^2. These bond tests can be shear, moving the two materials parallel to each other, or tensile, moving the two materials away from each other. The shear test is commonly used because of its ease in setup and speed. Unfortunately, it often results in cohesive failures because the stress distribution of the interface is inhomogeneous resulting in stress peaks that often initiate the fracture at the material and not at the interface, which is unacceptable (42) (43). Macrotensile strength testing involves separating the materials by moving them away from each other. This test applies a more even distribution of stresses on the materials. In 1994, Sano et al discovered that decreasing the surface size below 2 mm^2 gave a more homogenous interface which correlated to more adhesive failures (44). Therefore, micro-tensile bond tests have become a more preferred method of testing using areas of 1mm^2. The advantages of the micro-tensile bond test are that they permit testing of very small areas, produce more adhesive failures, higher initial bond strength, permits measurements of regional bond strengths, means and variances can be calculated for a single tooth, permits testing of bonds to irregular surfaces, and facilitates scanning electron microscopy examination of the failed bond (42). There are some disadvantages to this test that include: labor intensity that is highly demanding technically, requiring the use of specialized equipment sample integrity (i.e. drying out), and difficulty in measuring bond strengths less than 5 MPa (42).

Vitablocs Mark II, IPS e.max, Paradigm MZ100, and Lava Ultimate CAD/CAM Ceramics

Because of the new drive for esthetic crowns for the posterior teeth, there has been a large increase in production in different types of materials with several variations. Vitablocs Mark II (VITA Zahnfabrik) is an update to the Vitablocs Mark I (VITA Zahnfabrik), and is composed mostly of silicon dioxide (60-64%) and aluminum oxide (20-23%) at a particle size of 4 microns (45). The flexural strength is 150 MPa. The material is very brittle with a modulus of elasticity of 45 GPa. It has a survival rate 97% after five years, 95% after 9 years, and 84.4% after 18 years for inlays (45). The brand is marketed for its high esthetics and enamel like abrasion. They are available in the 10 most common Vita 3D – Master shades, and several types of multicolored blocks named TriLuxe blocks (45). It is one of the oldest CAD/CAM materials in use today and therefore is involved in most research involving CAD/CAM technology (46).

The IPS e. max (Ivoclar Vivadent) is an update to its predecessor, IPS Empress 2 (Ivoclar Vivadent), which was first released in 1998. The IPS e.max, released in 2006 is made of lithium disilicate material consisting of 1.5 microns making up 70% of the volume and suspended in a glass matrix (47). Manufacture states that their after processing flexural strength is 360 MPA, and the modulus of elasticity is 95 GPa (47). It is a glass ceramic so it is recommended to etch the material and silanate before cementing. Before final cementation the product needs to be fully crystallized. The crystallization step is unique to this product. Before final crystallization the material has a lower hardness and is easier to mill and adjust. The drawback is that the crystallization step takes an additional 20-30 minutes of appointment time (48). Since it's a relatively new product there are not a lot of long term studies performed on the material. The research that has been provided shows survivability of the product very close to 100% after 2 years (4), which shows very promising results in performance. The manufacturer has the blocks available in all the VITA shades along with high translucency, low translucency, and medium opacity. Giordano states that the material can obtain a high translucency because of the lithium disilicate's low refractive index (49). Della Bona, in 2006, compared the microtensile bond strength of Empress 2 and Empress 1. Both ceramics treated with hydrofluoric acid and silane had a bond strength of 31.9 (+/-8.6) MPa compared to Empress I's 26.4 (+/- 7.6) MPa. He stated that the difference can be explained by the reduced amount of silica in Empress 2. He concluded that the ceramic microstructure and ceramic surface treatment have a significant effect on the microtensile bond strength and failure mode (50).

Paradigm MZ100 (3M ESPE) was first introduced in 2000. It is considered a composite instead of a ceramic because it is a polymer composite block with zirconia-silica filler particles the average size of .6 microns making up 85% by weight embedded in a highly crossed-linked Bis-GMA-TEGDMA matrix (50). It was introduced as an alternative to a ceramic CAD/CAM. Giordano states that because of its composite make-up, it doesn't require firing or glazing, kinder to opposing natural dentition, easier finish and polish, and easier to make add-ons (46). The material has a flexural strength of 150 MPa and a flexural modulus of 21 GPa (51). Fasbinder states that these materials do not fracture easily under load because of their lower modulus of elasticity coupled with their flexural strength (52). Magne wants providers to consider preserving tooth structure by using the Paradigm MZ100 for occlusal veneers instead of using full coverage all-ceramics. He has demonstrated that the composite blocks have a significant increased fatigue resistance compared to the IPS Empress CAD and IPS e.max CAD (Ivoclar Vivadent) and are more able to withstand longterm functional loads (53). There are not very many long term survival studies, but Fasbinder et al. published a 10 year comparison between Paradigm MZ100 and Vitablocs Mark II inlays, which states that out of 80 blocks (40 Paradigm and 40 Vitablocs) Paradigm had a 95% survival rate compared to the Vitablocs 87.5% survival (54). Zohairy, in 2002, performed microtensile bond strengths between Vitablocs Mark II and Pardigm MZ1000 using different surface treatments and resin cements. The Vitablocs etched with hydrofluoric acid, silanated, and cemented with Nexus

2 cement had a bond strength of 24.3 (+/-3.1) MPa. The Paradigm MZ1000 treated the same and cemented with Nexus 2 had a bond strength of 54.5 (+/-6.9) MPa. He stated that the difference in bond strength was strongly correlated to the material's modulus of elasticity (3).

Released in 2011, a resin nano ceramic material, Lava Ultimate (3M ESPE), uses a highly crossed linked bisphenol -A resin matrix which makes up 80% by weight. Embeddded in the matrix are aggregated particles of silica and zirconia. Silica particles average 20 nanometers and the zirconia are 4-11 nanometers (55). The flexural strength is 200 MPa. The material does not require firing. Compared to other ceramic materials, the Lava Ultimate has greater edge stability because its modulus of elasticity is 12.77 GPa (55) (56). Since its recent arrival to the dental world it has little research behind it. The material is similar to the 3M Paradigm which is zirconia-silica embedded in a resin matrix (55). An advantage that the resin nano ceramic has over the composite blocks is that it is able to retain a high-gloss surface finish over time because of it ceramic fillers (57) (58).

There are many factors that are present in the use of CAD/CAM all-ceramic restorations. There are several different manufacturers that produce material that have their advantages and disadvantages. There is no ideal restoration or cement. It is up to the provider to know the details of the materials to accurately weigh the risks and benefits to provide the best treatment. Multiple studies have reported that composite blocks have higher microtensile bond strength because of their lower elastic modulus (12.77-21 GPa) (51) (3). Observing the properties of the Lava Ultimate and the limitations within a tensile bond strength test, it is hypothesized that the material with such a low modulus of elasticity will have a higher tensile strength compared to the feldspathic Vitablocs Mark II reflecting its ability to resist radial fractures and increasing its survival.

PURPOSE

The purpose of this study is to investigate the microtensile bond strength of resin cement bonded to composite and ceramic CAD/CAM materials.

HYPOTHESIS

Hypothesis: CAD/CAM resin nano ceramic has a higher bond strength compared to feldspathic ceramic.

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SPECIFIC AIMS

Specific aim 1: Determine the microtensile bond strength of resin cement to CAD/CAM nano resin ceramic.

Specific aim 2: Determine the microtensile bond strength of resin cement to CAD/CAM feldspathic ceramic.

Specific aim 3: Compare the microtensile bond strength between CAD/CAM resin nano ceramic and CAD/CAM feldspathic ceramic.

MATERIALS AND METHODS

Bonding the composite to the CAD/CAM blocks

Ceramic and resin nano ceramic blocks, which are designed for the CEREC CAD/CAM system (Sirona, Charlotte, NC), are used for the study. Four CEREC Vitablocs MarK II (fine-particle feldspar ceramic blocks) I14, size 12x14x18 mm^3, (Vita, Bad Sackingen, Germany), and four 3M Lava Ultimate (resin nano ceramic) size 14L (3M ESPE Saint Paul, MN) were used. Lava Ultimate block was cut 1 mm from the 12x14 end of the block parallel to the end. The area was sandblasted with aluminum oxide grain ≤ 50 microns (Renfert; St. Charles, IL) until the entire bonding surface appeared matted. The block was rinsed with tap water for 1 minute and then ultrasonically cleaned with distilled water for 5 minutes. The blocks were dried with compressed air for one minute. Nexus 3 (Kerr; Orange, CA) silane was placed and air dried for one minute.

The Vitablocs MarK II blocks were cut similarly to the Lava Ultimate block, but not sandblasted. Hydrophloric acid 9.5% was applied for one minute, rinsed for one minute with tap water and then placed in ultrasonic cleaner with distilled water for five minutes. The blocks were dried with compressed air for 1 minute. Nexus 3 silane was placed and air dried for one minute.

For all the blocks, a thin layer of Nexus 3 light cured cement was dispensed with auto mixed syringe and applied with a microbrush. Thin layer was light cured with Adec chairside curing light (Adec; Deerfield, IL) for 10 seconds. Z100 composite, shade A3 (3M ESPE Saint Paul, MN) was layered in 2 mm increments, up to 5mm, and was light cured for one minute between increments.

Preparing the 1mm^2 slabs from the blocks

Blocks were cut using a low speed cutting saw (Beuhler Ltd, Lake Bluff, IL). , Initial set of cuts making 1mm slabs were made perpendicular to the adhesive interface starting at the composite and moving towards the ceramic. The cuts were made >6 mm into the ceramic. The block was rotated 90 degrees and a second set of 1mm slab cuts made perpendicular to the adhesive interface starting at the composite and moving to the ceramic so that there are resulting 1 mm^2 slabs. The slabs were separated at the ceramic base with mild stress using thin dental instrument. Slabs were randomly observed under the 30x microscope for any obvious defects from cutting and measured with a digital calibrator to confirm the sizes were 1mm^2(+/-0.5). Twelve slabs were randomly selected from each block. The slabs were stored in distilled water at 37°C for 24 hours.

Applying the tensile strength

Slabs were bonded in a vertical direction on Instron universal testing plates with cyanoacrylate and placed in a universal testing machine (Model no. 5943 ; Instron; High Wycombe, Bucks, UK)under tensile stress with a crosshead speed of 1mm/min until failure. Micro-tensile bond strength calculations were made using the following equation: s = L / A, where “s” is the bond strength

(MPa), “L” = test load (N), “A” = adhesive area (mm^2). To determine the mode of failure, slabs were viewed under a 30x microscope.

Failure modes were categorized as one of the following types: A= Adhesive failure at the cement-ceramic interface; B= Cohesive failure in the ceramic; C= Cohesive failure mode in the cement; D= mixed A and B; and E= Mixed A and C. Since, the study is focused on micro-tensile bond strength of the ceramic material, only type A failure were acceptable for data collection. Two-Factor ANOVA test was performed to compare the failures within the block types and between the block types.

RESULTS

From the data gathered, the Vitablocs Mark II showed a mean microtensile bond strength of 18.5 (+/-3.7) MPa. The Lava Ultimate had a mean bond strength of 47.6 (+/- 12.3). The blocks means were separated into four groups labeled ‘A’, ‘B’, ‘C’, and ‘D’. The mean bond strength values of Vitablocs Mark II were: **A**: 19.3(+/- 4.5)MPa, **B**:20.9(+/-3.8)MPa, **C**: 16.9(+/-3.1), and **D**:16.7(+/-3.4). The mean bond strength values of Lava Ultimate were: **A**: 54.3(+/- 24.0)MPa, **B**: 49.1(+/- 9.9)MPa, **C**: 44.1(+/-8.2)MPa, and **D**: 42.8(+/-6.8)MPa. Comparing the two different ceramics statistically using Two-Factor Analysis of Variance test (ANOVA) and Tukey’s Studentized Range test, the μTBS between the Vitablocs Mark II and Lava Ultimate were statistically questionable (P=0.052 with confidence > 95%). Since the P value was greater then 0.052 and the author believes this to be at the fault of the author’s inexperience with the experiment protocol. The ANOVA test was reworked with the first Lava Ultimate and Vitabloc Mark II omitted. The P value was lowered to 0.011 (Confidence > 95%), which is a significant statistical difference.

DISCUSSION

After review of the literature, a main proponent to ceramic fracture is the bond strength to the tooth. Bond tests are a good measurement to help identify the durability of the ceramic materials under load (40). The μTBS was greater with the Lava Ultimate. These findings are consistent with CAD/CAM resin blocks and feldspathic blocks from a previous study by Zohairy (3), yet there has been no research to this author's knowledge that has tested the bond strength of Lava Ultimate. It is easy to speculate that the bond strength would be greater because the resin cement is being applied to a block which is comprised mostly of resin. Zohairy,stated that the higher bond strength is related to the mechanical differences between the composite and the ceramic. The elastic modules between the two materials possibly make the difference. The higher the elastic modulus of the material, the higher the stresses generated at the edge of the bonding interface (3). Similar statement was reported by Bella Dona in his 2006 study: "Apparent interfacial fracture toughness of resin/ceramic systems" (51). The Lava Ultimate has an elastic modulus of 12.77 GPa (55) and the Vitabloc Mark II has an elastic modulus of 95 GPa (45).

The most significant factors to performing the μTBS are the technique sensitivity and labor intensity. From preparing the blocks to applying the tensile stress there are many points at which error can be introduced and the results can be skewed. Therefore, it is important to have a protocol that the operator understands and has practiced multiple times with products of a known tensile strength so that there is less questioning of the results.

The results showed that there was a difference in bond strengths between the Vitablocs MarK II and the Lava Ultimate, but there were significant differences within the Lava Ultimate. It appeared evident to the investigator looking at the results from the beginning to end, that the standard deviations began to become narrower. The investigator reasons this to technical challenges in performing the test. What were the major factors for this ‘tightening groups’?

1. **Treating the surface of the blocks prior to applying the cement**

This was first major step where differences were seen. Reviewing the figures 3 and 4 of the surface treated blocks, the Vitablocs MKII appear to have similar pattern of etching. On the other hand, the Lava Ultimate blocks are a resin matrix and not glass, and therefore recommened to be sandblasted by manufacturer guidelines. Giving a uniformed surface treatment is difficult when the sandblasting device projects in a stream. Placing the sandblaster too close or for too long in one section will remove more of the block creating an irregular surface. Looking at the figure it is evident that the investigator over-blasted block ‘A’ and over-corrected by under-blasting block ‘D’. Zohairy avoided this problem in their 2002 study by grinding the composite blocks on a polishing machine with 600 grit SiC paper (3).

2. **Damage to the slabs while sectioning**

As viewed in the figures, it is clear to see that the sectioning of the blocks into 1mm^2 slabs has a potential to alter the slabs and give a inaccurate result.

The slabs were viewed under a 30x microscope before and after applying tensile forces, but crack formation may not be detectable at the level and may need more enhanced viewing equipment such as SEM or stereomicroscope, which was not available. Della Bona recommends a thorough SEM examination of the materials and fractured surfaces. He also recommends comfirmation of the restorative material's composition through x-ray elemntal map analysis. Both of these ensure a more consistent and complete description of the fracture process and mode of failure (51). All slabs with known defects were not used for this experiment.

3. **Speed and expertise with using Instron Universal Tester.**

When performing the bond strength tests the protocol begane the Lava Ultimate 'A' and worked progressively through to Vitabloc MKII 'D'. It is within the authors thoughts that moisture content contributes to crack propagation and failure of the bond. Kassem et al, in a 2012 study stated that a stress dependent chemical reaction occurs between water and surface flaws in the crown resulting in their growing to a critical size (29). Attia also makes a similar statement in a 2012 study (34). The slabs are very thin and short and therfore do not hold moisture for long. The longer it takes to mount the slab and perform the test, the drier the specimens become. By the time the Vitablocs MarKII specimens where being tested, the user was profecient with the protocol.

Since there was a significant deviation within the first Lava Ultimate block due to operator's experience with protocol, the first tested blocks of the Lava

Ultimate and Vitabloc MKII were left out of the second ANOVA test. The P value decreased from 0.052 wich is a questionable statitistical difference to a 0.011, which is statistically significant difference.

CONCLUSION

The null hypotheseis of this experiment was that there was no difference between the μTBS of Vitabloc MKII and Lava Ultimate CAD/CAM ceramics was rejected. There is a statistically significant difference and therefore the alternative hypothesis that there is a difference should be accepted. Within the limitation of this study, the bond strength of the Lava Ultimate CAD/CAM nano resin ceramics, were shown to be greater than that of feldspathic Vitablocs Mark II. Further research involving more robust studies, such dynamic loading and cyclic fatigue testings along with clinical longivity need to be performed with the Lava Ultimate to show its capabilities and limitations to aid clinicians in their case selection for treatment planning and execution.

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TABLES

Vitabloc MKII (1A)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.81	0.79	15.77361
2	0.82	0.96	25.02001
3	0.87	0.86	14.9265
4	0.87	1.02	15.00573
5	1.08	0.73	15.08161
6	0.81	0.99	20.95042
7	0.99	0.96	21.0435
8	1.08	0.96	21.34102
9	1.03	0.85	16.2625
10	1.23	0.88	15.36007
11	0.99	0.72	27.83158
12	0.98	0.95	22.80906
Maximum	1.23	1.02	27.83158
Mean	0.96333	0.88917	19.2838
Median	0.985	0.915	18.60646
Minimum	0.81	0.72	14.9265
Standard deviation	0.13124	0.10104	4.47512
Mean + 1 SD	1.09457	0.9902	23.75892
Mean - 1 SD	0.83209	0.78813	14.80868

Table 1. Raw data of the Vitabloc MKII ‘A’ μTBS test

Vitabloc MKII (1B)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.86	0.83	25.47625
2	0.93	0.9	17.27497
3	1.03	0.83	23.60264
4	0.88	1.13	26.88652
5	1	1.07	22.20883
6	1.03	0.9	19.39349
7	0.99	1.01	13.64077
8	0.9	0.83	18.19075
9	0.85	0.98	23.59552
10	1.05	0.83	19.44482
11	1.03	0.92	19.12377
12	0.99	0.84	22.54753
Maximum	1.05	1.13	26.88652
Mean	0.9617	0.9225	20.94882
Median	0.99	0.9	20.82682
Minimum	0.85	0.83	13.64077
Standard deviation	0.0733	0.1035	3.77153
Mean + 1 SD	1.035	1.026	24.72035
Mean - 1 SD	0.8883	0.819	17.17729

Table 2. Raw data of the Vitabloc MKII ‘B’ μTBS test

Vitabloc MKII (1C)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.93	0.95	18.94961
2	0.93	0.94	13.85358
3	0.95	1.02	13.34775
4	0.93	0.95	15.99494
5	0.95	0.92	21.07936
6	0.93	0.97	20.42967
7	0.95	0.94	12.79231
8	0.99	0.91	20.01138
9	0.91	0.94	20.11421
10	0.71	0.95	16.14097
11	0.95	0.98	14.0019
12	0.93	0.73	15.98607
Maximum	0.99	1.02	21.07936
Mean	0.92167	0.93333	16.89181
Median	0.93	0.945	16.06795
Minimum	0.71	0.73	12.79231
Standard deviation	0.06952	0.07011	3.06801
Mean + 1 SD	0.99119	1.00344	19.95982
Mean - 1 SD	0.85214	0.86323	13.8238

Table 3. Raw data of the Vitabloc MKII ‘C’ μTBS test

Vitabloc MKII (1D)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	1.01	1.06	14.3073
2	1	0.99	16.3527
3	1.07	1.16	13.06218
4	0.77	1.17	18.31256
5	0.88	1.09	10.61889
6	1.07	0.97	19.4865
7	1.08	0.79	18.0826
8	0.88	0.94	18.07461
9	1.03	1.03	19.41893
10	1.04	0.88	12.40372
11	0.96	0.93	20.65668
12	1	0.9	20.20956
Maximum	1.08	1.17	20.65668
Mean	0.9825	0.9925	16.74885
Median	1.005	0.98	18.0786
Minimum	0.77	0.79	10.61889
Standard deviation	0.09459	0.11458	3.35775
Mean + 1 SD	1.07709	1.10708	20.10661
Mean - 1 SD	0.88791	0.87792	13.3911

Table 4. Raw data of the Vitabloc MKII ‘D’ µTBS test

Lava Ultimate (2A)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.95	0.84	19.23048
2	0.91	0.94	31.07017
3	0.96	0.96	18.88048
4	0.91	0.98	48.06977
5	1.01	0.98	81.60197
6	0.93	0.96	92.18764
7	0.87	0.92	52.33662
8	0.95	0.94	66.72845
9	1.2	0.92	37.82509
10	0.84	0.92	63.91354
11	0.93	0.92	76.81454
12	0.96	0.89	63.14515
Maximum	1.2	0.98	92.18764
Mean	0.95167	0.93083	54.31699
Median	0.94	0.93	57.74089
Minimum	0.84	0.84	18.88048
Standard deviation	0.08983	0.03942	24.02844
Mean + 1 SD	1.0415	0.97025	78.34544
Mean - 1 SD	0.86184	0.89142	30.28855

Table 5. Raw data of the Lava Ultimate ‘A’ μTBS test

Lava Ultimate (2B)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.97	1.06	23.87563
2	0.9	0.87	55.2632
3	0.94	0.87	54.00829
4	0.99	0.76	40.89864
5	0.92	0.74	51.94327
6	0.95	0.92	62.96262
7	0.97	1.03	49.84886
8	1.03	0.97	49.04372
9	0.92	0.9	48.9954
10	1.14	0.93	50.66461
11	0.93	0.99	57.81736
12	1.01	1	44.26457
Maximum	1.14	1.06	62.96262
Mean	0.9875	0.92	49.70126
Median	0.97	0.925	50.25673
Minimum	0.92	0.74	23.87563
Standard deviation	0.06797	0.09954	9.9859
Mean + 1 SD	0.05547	1.01954	59.28716
Mean - 1 SD	0.91953	0.82046	39.3536

Table 6. Raw data of the Lava Ultimate ‘B’ μTBS test

Lava Ultimate (2C)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.94	0.89	45.70728
2	0.91	0.87	43.9316
3	1.07	0.87	36.36125
4	0.89	0.81	53.0699
5	0.9	0.88	27.80654
6	0.86	0.95	50.01054
7	0.97	0.93	57.63116
8	1	1	47.32933
9	1	1	46.57085
10	1	1	38.54042
11	0.87	0.92	36.17879
12	0.95	0.89	46.06161
Maximum	1.07	1	57.63116
Mean	0.94385	0.91308	42.56493
Median	0.94	0.89	45.70728
Minimum	0.86	0.81	24.1448
Standard deviation	0.06172	0.06033	9.60935
Mean + 1 SD	1.00556	0.97341	52.17428
Mean - 1 SD	0.88213	0.85275	32.95558

Table 7. Raw data of the Lava Ultimate ‘C’ μTBS test

Lava Ultimate (2D)			
	Width	Thickness	Tensile stress at Break (Standard)
	(mm)	(mm)	(MPa)
1	0.9	0.95	48.67526
2	1	0.89	44.50586
3	0.93	1.06	33.30247
4	0.94	0.92	50.77501
5	0.85	0.91	48.57327
6	0.9	0.81	38.53666
7	1.01	0.94	49.05416
8	0.86	0.94	42.41837
9	0.91	0.85	33.30345
10	0.89	0.91	45.20741
11	0.9	0.88	46.88523
12	1.1	0.88	32.47256
Maximum	1.1	1.06	50.77501
Mean	0.9325	0.91167	42.80914
Median	0.905	0.91	44.85663
Minimum	0.85	0.81	32.47256
Standard deviation	0.07149	0.06162	6.75091
Mean + 1 SD	1.00399	0.97329	49.56006
Mean - 1 SD	0.86101	0.85005	36.05823

Table 8. Raw data of the Lava Ultimate 'D' μTBS test.

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	7	21492.25362	3070.32195	29.16	<.0001
Error	88	9265.41932	105.28886		
Corrected Total	95	30757.67294			

R-Square	Coeff Var	Root MSE	TSB Mean
0.698761	31.06680	10.26104	33.02894

Source	DF	Anova SS	Mean Square	F Value	Pr > F
Type	1	20353.12119	20353.12119	193.31	<.0001
SEQ	3	845.97123	281.99041	2.68	0.0519
Type*SEQ	3	293.16120	97.72040	0.93	0.4307

Table 9. 2-Way ANOVA Tables of Mean μTBS for Vitabloc MKII and Lava Ultimate blocks ‘A-D’. ‘SEQ’= Mean distirbution between Lava Ultimate and and Vitabloc MKII; ‘Type*SEQ’= Slabs with the blocks.

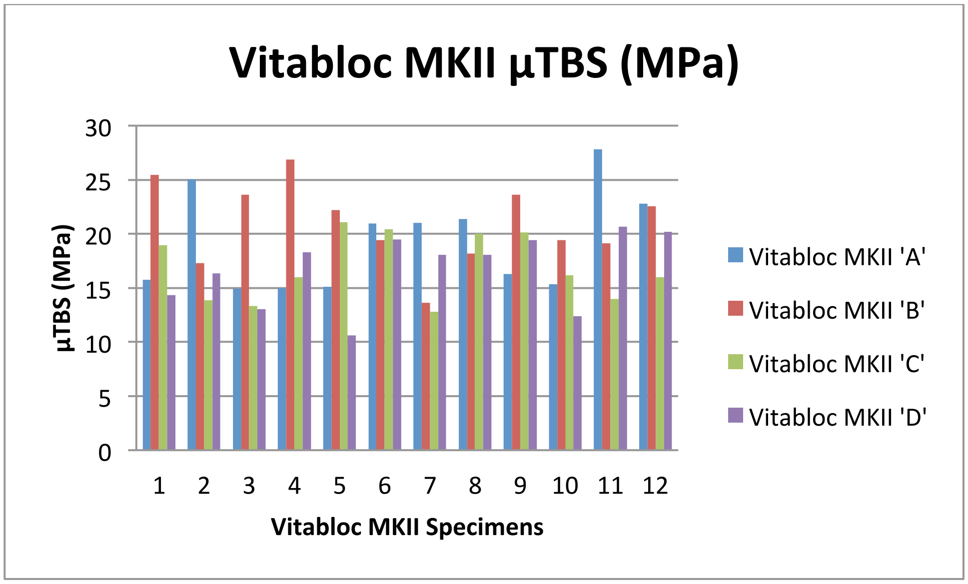
Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	5	13673.14328	2734.62866	66.99	<.0001
Error	66	2694.09820	40.81967		
Corrected Total	71	16367.24149			

R-Square	Coeff Var	Root MSE	TSB Mean
0.835397	20.10912	6.389027	31.77179

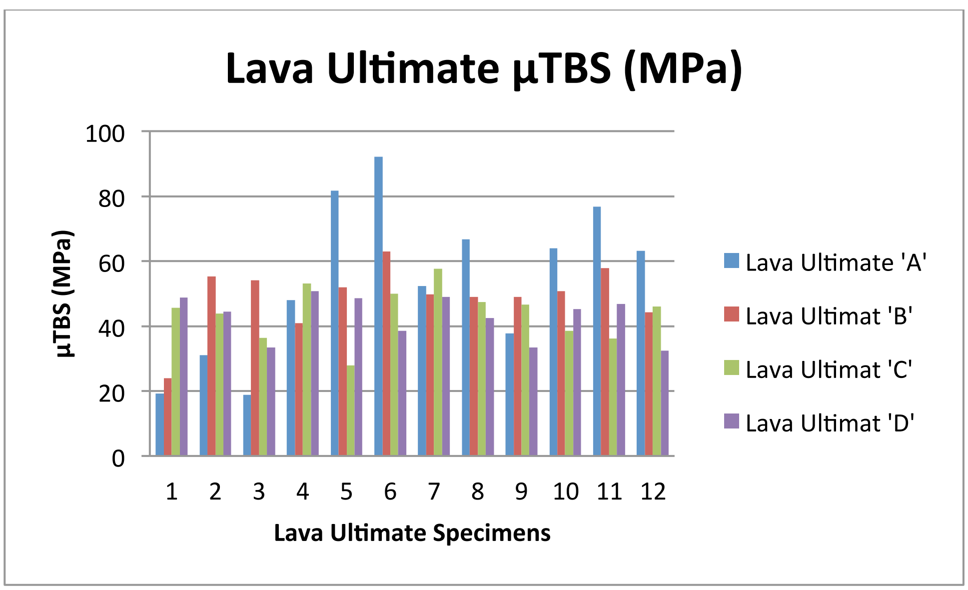
Source	DF	Anova SS	Mean Square	F Value	Pr > F
Type	1	13268.78357	13268.78357	325.06	<.0001
SEQ	2	390.80765	195.40383	4.79	0.0114
Type*SEQ	2	13.55206	6.77603	0.17	0.8474

Table 10. 2-Way ANOVA Tables of Mean µTBS for Vitabloc MKII and Lava Ultimate blocks ‘B-D’. ‘SEQ’= Mean distirbution between Lava Ultimate and and Vitabloc MKII; ‘Type*SEQ’= Slabs with the blocks.

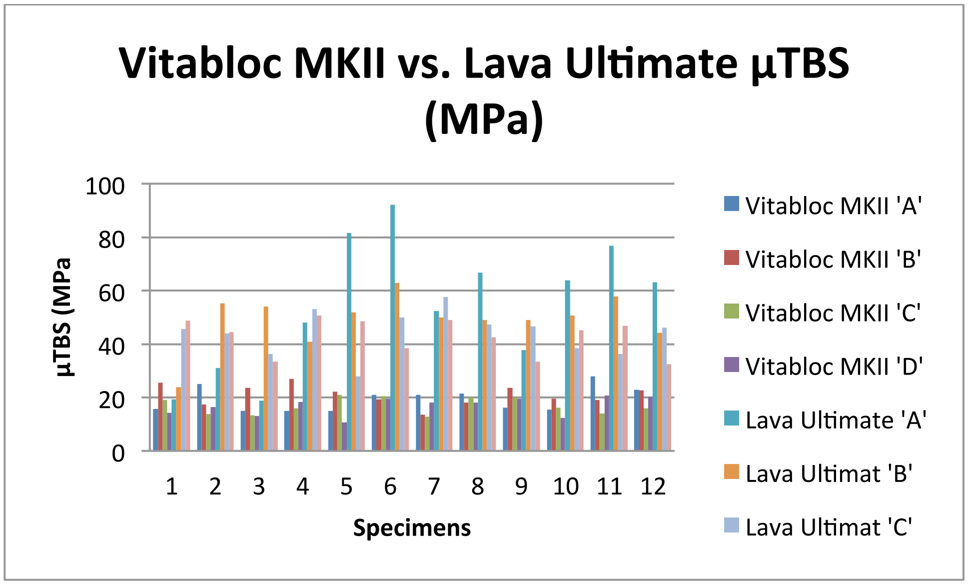
GRAPHS



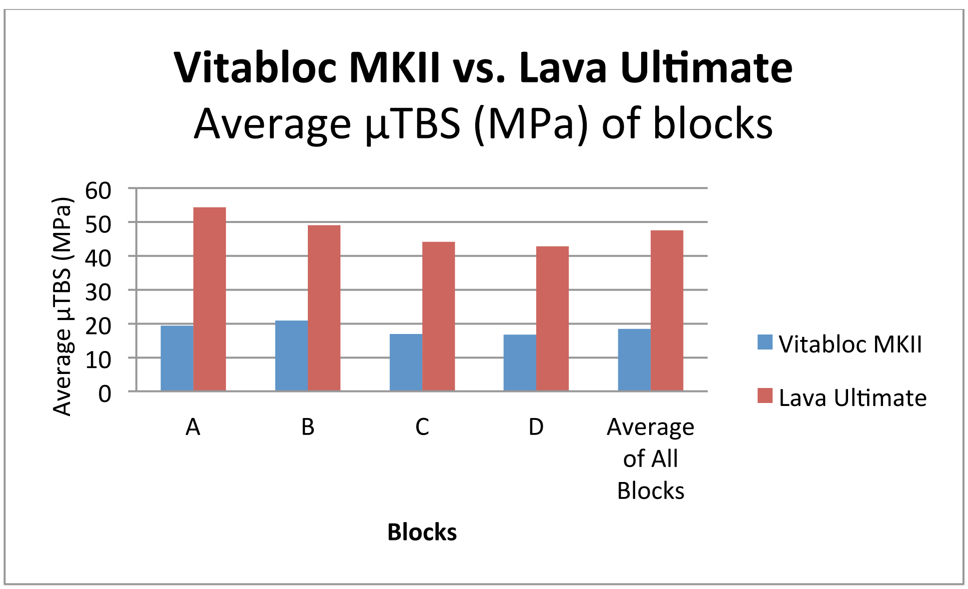
Graph 1. Results of the Vitabloc MKII µTBS test



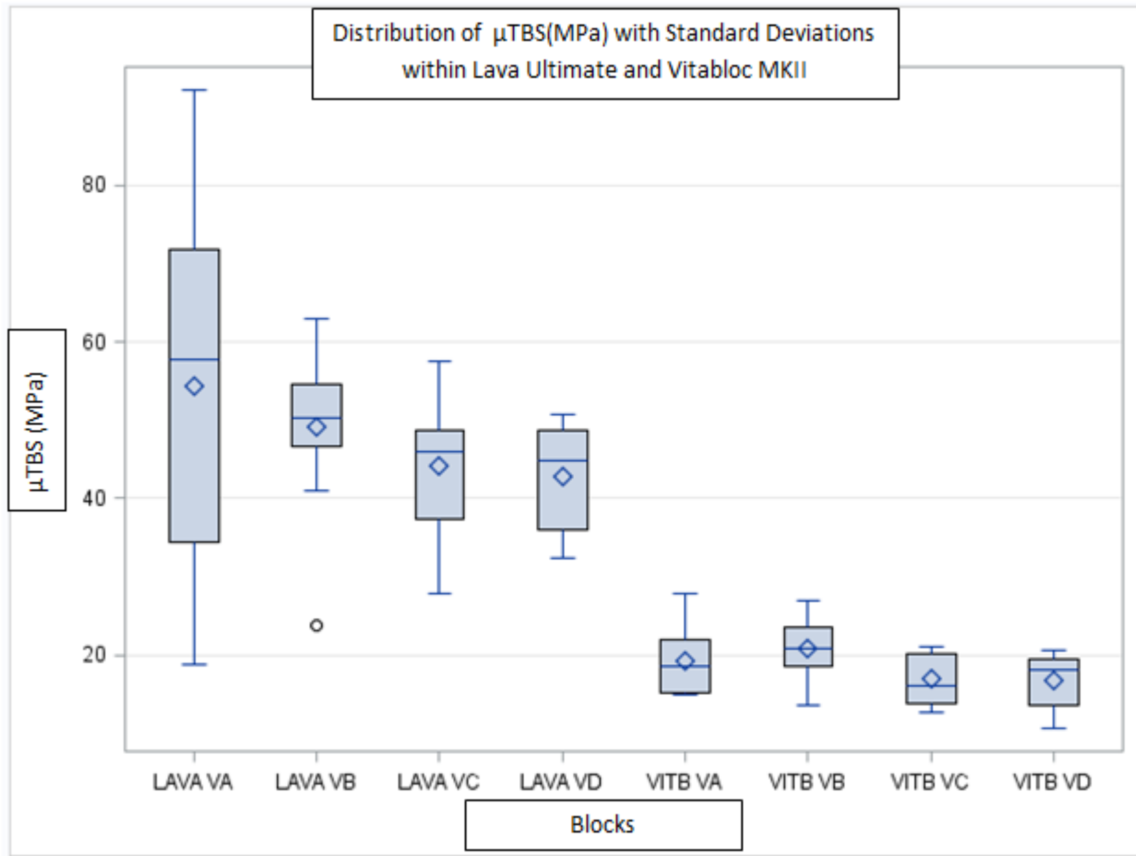
Graph 2. Results of the Lava Ultimate µTBS test



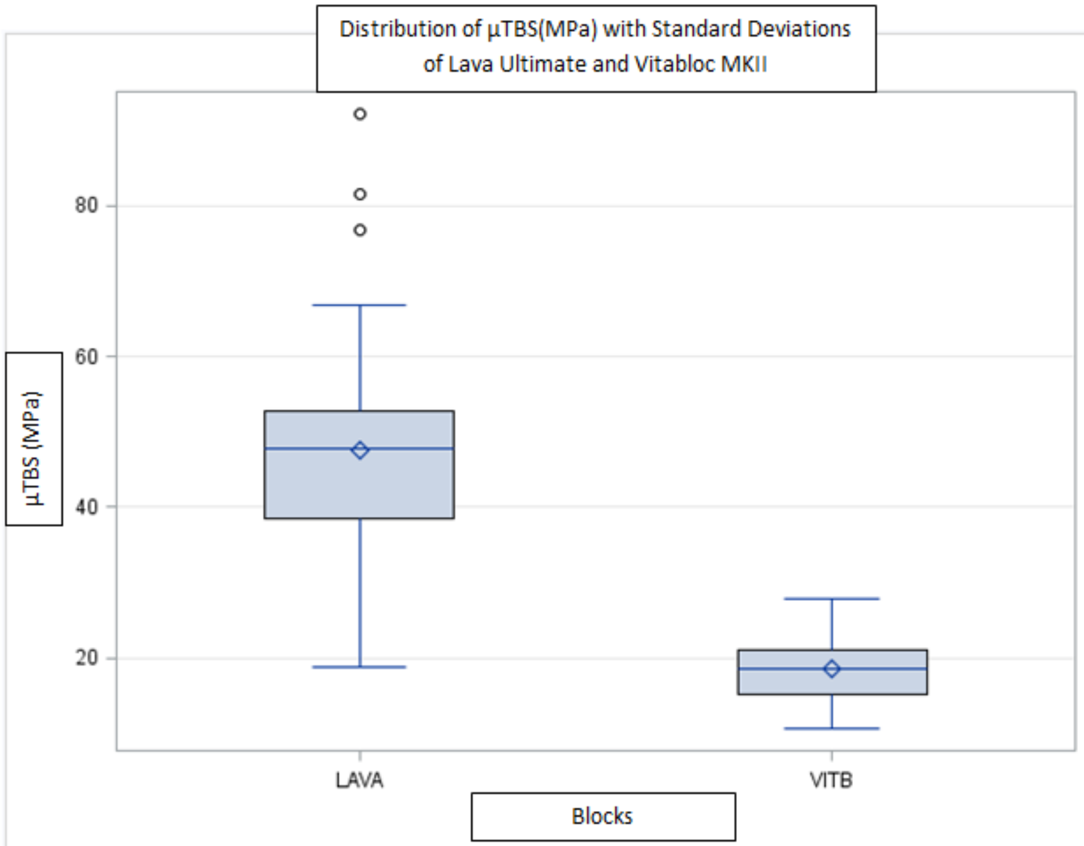
Graph 3. Compared results of the Vitabloc MKII and Lava Ultimate µTBS



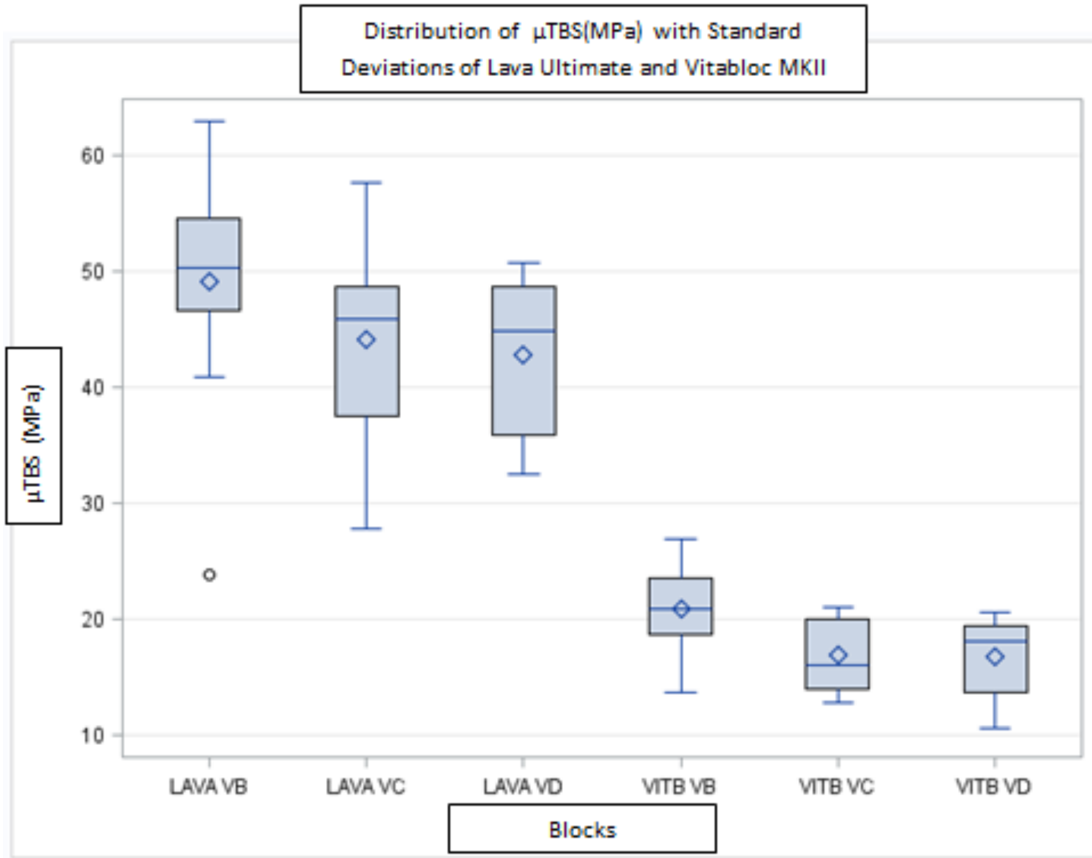
Graph 4. Compared means of the Vitabloc MKII and Lava Ultimate µTB.



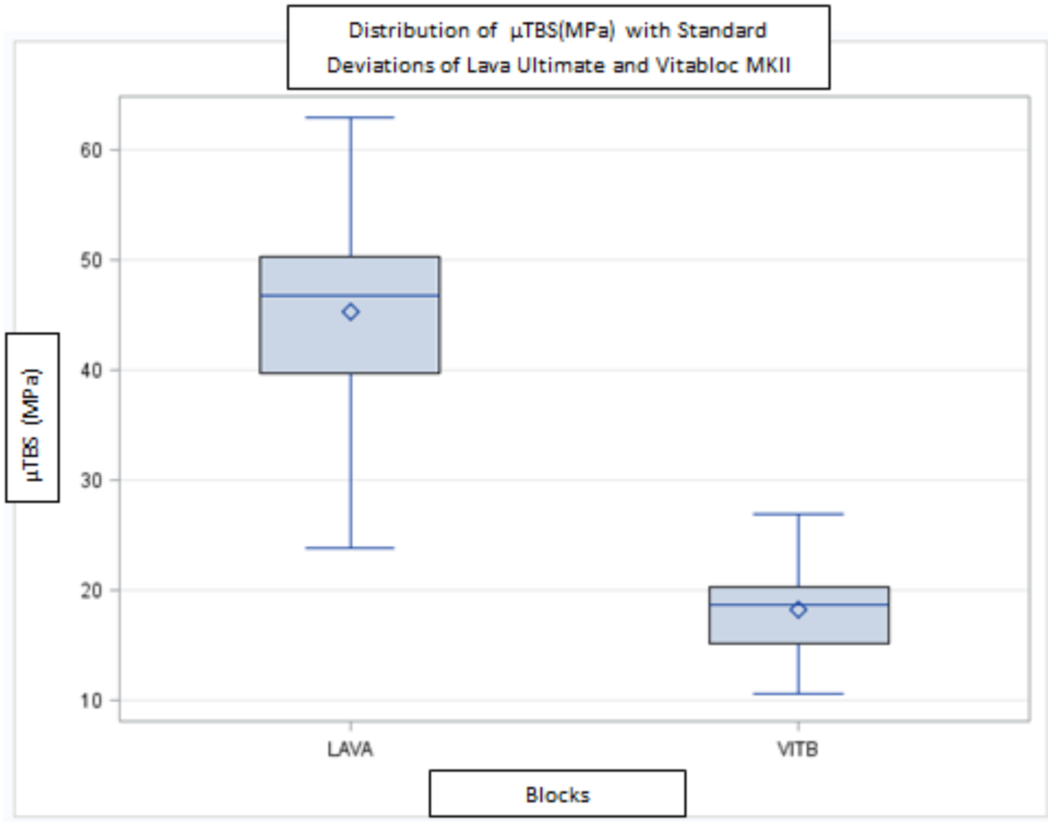
Graph 5. Distribution of mean µTBS within the blocks 'A-D'. The mean value of the blocks were not statistically different within the Vitabloc MKII or Lava Ultimate *(P* value > 0.05).



Graph 6. Distribution of mean µTBS of the blocks 'A-D'. The mean values between the Vitabloc MKII and the Lava Ultimate had a questionable statistical difference (*P* value = 0.052)



Graph 7. Distribution of mean µTBS within the blocks 'B-D'. The mean value of the blocks were not statistically different within the Vitabloc MKII or Lava Ultimate *(P* value > 0.05, Confidence >95%).



Graph 8. Distribution of mean µTBS of the blocks 'B-D'. The mean values between the Vitabloc MKII and the Lava Ultimate were statistically different (*P* value = 0.011, Confidence > 95%)

PHOTOS

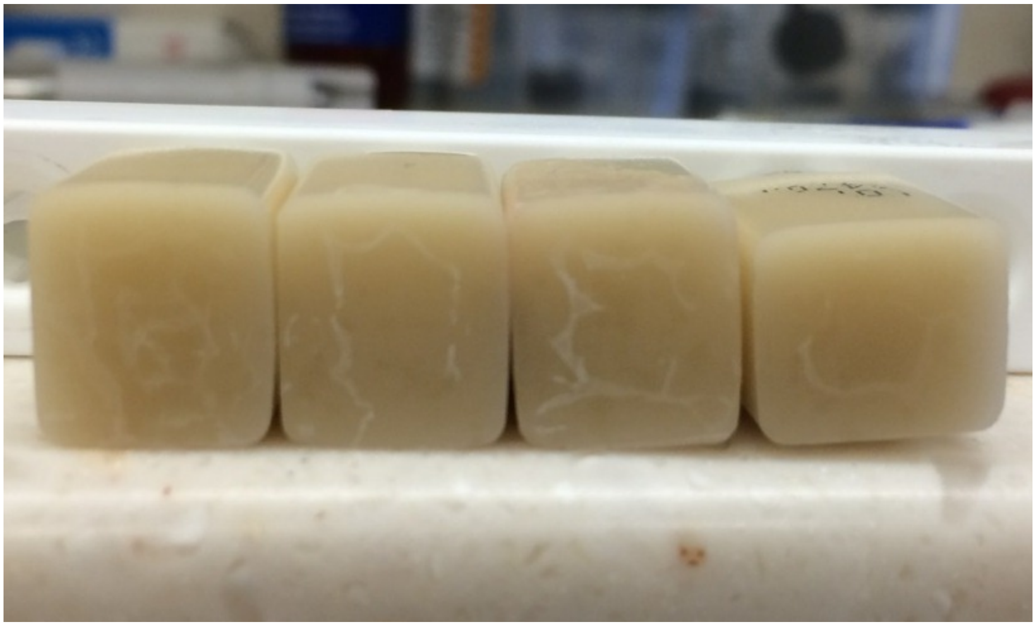


Figure 3. Vitabloc MKII blocks etched with hydrofluoric acid.

Figure 4. Lava Ultimate blocks air abraded.

Figure 5. Lava Ultimate with a thin layer of cement.

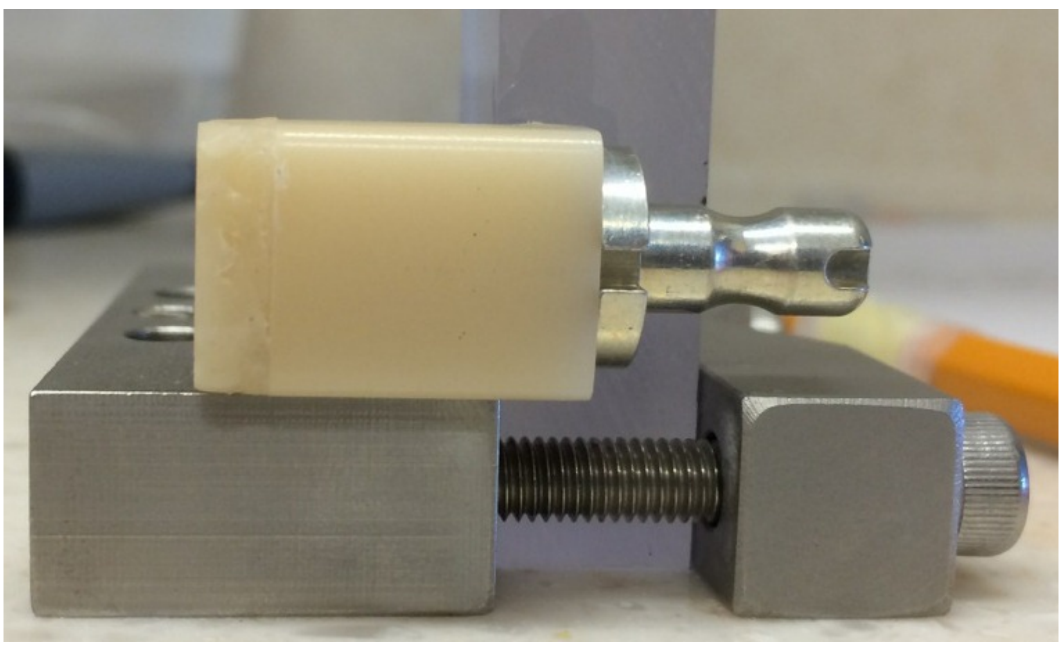


Figure 6. Block placed in sectioning jig.

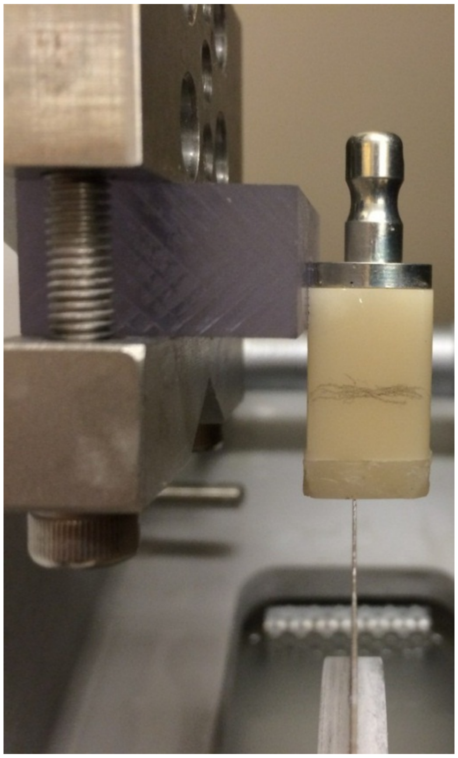


Figure 7. Block being sectioned with low-speed saw.

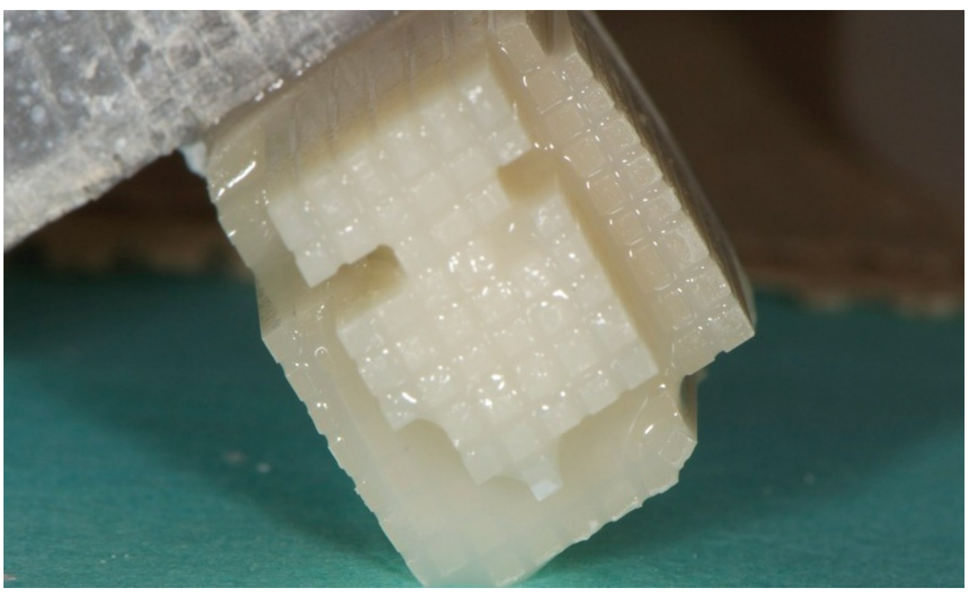


Figure 8. Slabs that have been lost or premature debonding during sectioning of the Vitabloc MKII. This was not evident with the Lava Ultimate blocks.

Figure 9. 1mm^2 Slab with defect from sectioning.

Figure 10 and11. Instron 5843 Universal Testing Machine and μTBS jig.

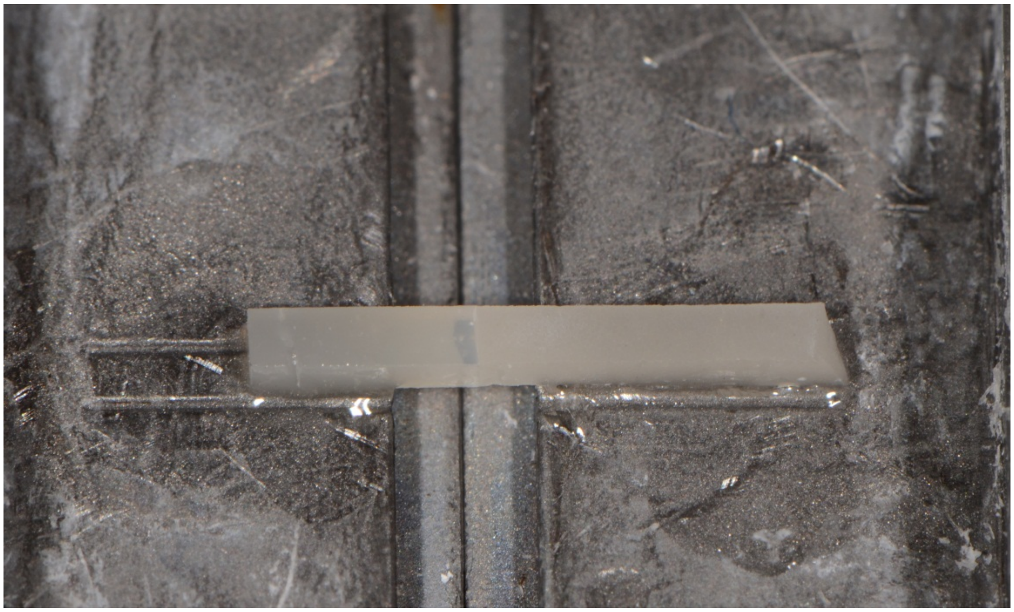


Figure 12. Lava Ultimate 1mm^2 slab with Z100 composite on left, cement layer in the middle, and Lava Ultimate on right.

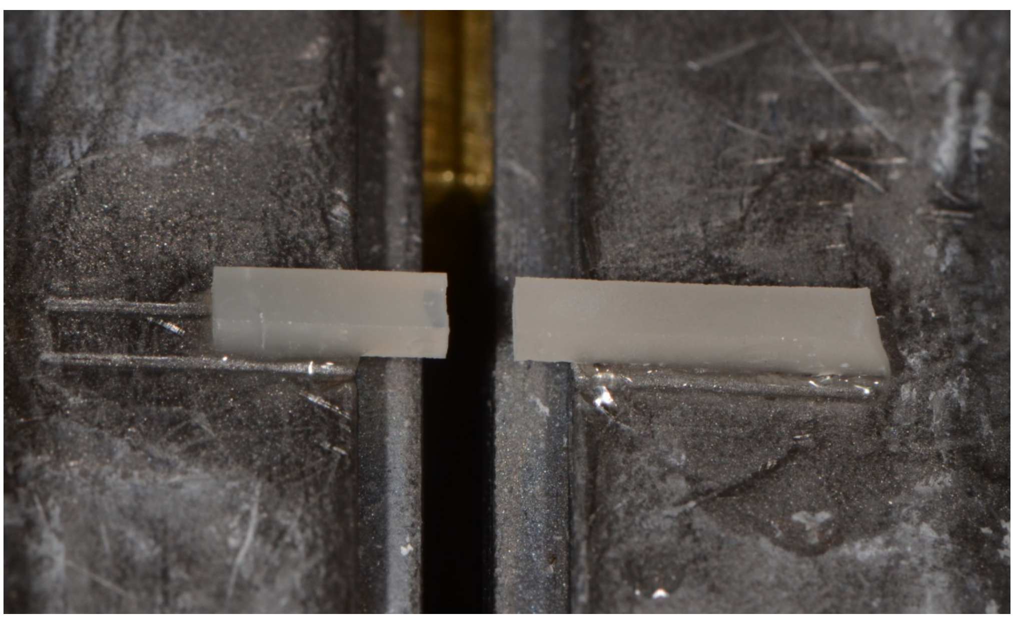


Figure 13. Lava Ultimate 1mm^2 slab demonstrating adhesive failure at the ceramic/cement interface.

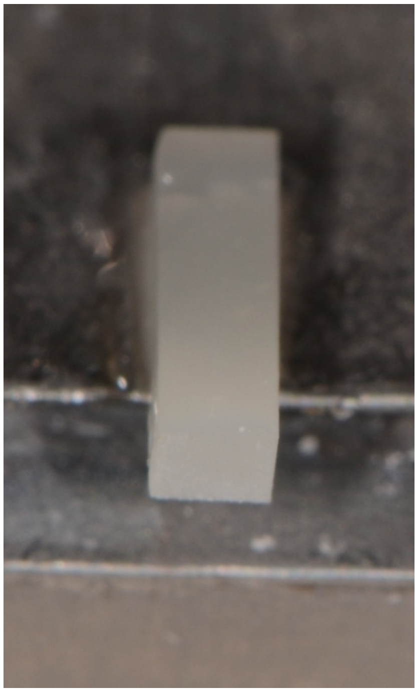


Figure 14. View of separated Lava Ultimate surface showing the adhesive failure.