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Static and cyclic loading effects on fracture toughness of contemporary CAD/CAM restorative materials

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DISSERTATION

STATIC AND CYCLIC LOADING EFFECTS ON FRACTURE TOUGHNESS OF CONTEMPORARY CAD/CAM RESTORATIVE MATERIALS

by

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ABSTRACT

OBJECTIVES

To test and compare the effects of static and cyclic loading on fracture toughness (K_{1C}) and microhardness of dental restorative CAD/CAM materials.

MATERIAL AND METHODS

Five commercially available CAD/CAM restorative materials were included in this study: Lava™ Ultimate Restorative (3M ESPE), IPS Empress® CAD (Ivoclar Vivadent), Enamic® (VITA), IPS e.max® CAD (Ivoclar Vivadent), and CERASMART™ (GC Dental). Polished rectangular bars 4×2×14 mm (n=30) were prepared from mill blocks for each material. Single notch of 0.5-1 mm in depth was made on the center of one length edge. Ten specimens per group for each material were randomly selected for 1) static mode, 2) after 100k cyclic loads, and 3) after 200k cyclic loads. The survival bars after the fatigue test were then subjected to a three-point flexural test. K_{1C} values were

determined on ‘single-edge-pre-crack-beams’ (SEPB) method. In addition, random specimens after the flexural test were selected for Vickers microhardness test from each group. Additionally indentation fracture method (IF) was used to determine surface fracture toughness for e.max CAD and Empress CAD. All the results were analyzed via ANOVA with Tukey’s HSD test or least square regression model using JMP Pro 12.0.

RESULTS

The mean fracture toughness (K_{1C}) of the material tested in static mode (3.2 $MPa{\cdot}m^{1/2}$ for e.max CAD, 2 $MPa{\cdot}m^{1/2}$ for Lava Ult, 1.95 $MPa{\cdot}m^{1/2}$ for Empress CAD, 1.92 $MPa{\cdot}m^{1/2}$ for Enamic, and 1.65 $MPa{\cdot}m^{1/2}$ for Cerasmart).

The 100k fatigue group (4.02 $MPa{\cdot}m^{1/2}$ for e.max CAD, 3.06 $MPa{\cdot}m^{1/2}$ for Cerasmart, 2.55 $MPa{\cdot}m^{1/2}$ for Lava Ult, 2.01 $MPa{\cdot}m^{1/2}$ for Enamic, 1.94 $MPa{\cdot}m^{1/2}$ for Empress CAD)

The 200k fatigue group (3.14 $MPa{\cdot}m^{1/2}$ for Cerasmart, 2.83 $MPa{\cdot}m^{1/2}$ for Lava Ult, 2.68 $MPa{\cdot}m^{1/2}$ for e.max CAD, 2.01 $MPa{\cdot}m^{1/2}$ for Enamic, 1.72 $MPa{\cdot}m^{1/2}$ for Empress CAD).

While there was a significant difference in the mean fracture toughness (K_{1C}) and (VHN) after fatigue of material tested ($p<0.05$).

CONCLUSION

The CAD/CAM materials tested exhibited a higher K_{1C} values after cyclic loading, along with lower K_{1C} compared to the static group. In addition, K_{1C} values by IF method exhibit lower K_{1C} values after fatigue that was not a good way to test the fracture toughness value.

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Chapter 1. INTRODUCTION

Dentistry is a field that develops day after day, with new materials and new technologies. In the past few decades there was a tremendous change in the quality of materials and new techniques for both dentist and technicians.

Computer-aided design (CAD) and computer-aided manufacturing (CAM) have increasingly become a very popular tool in the dental arsenal, not only for the dentists but also for patients. The comprehensive application of CAD/CAM technology is taking place due to its multiple advantages.

1.1 CAD/CAM Brief historical background

The CAD/CAM technology concept was introduced in dentistry by Dr. Duret in the early 1970s when he developed the first dental CAD/CAM device.(1) Dr. Duret introduced the first CAD/CAM dental restoration in 1983 and in 1985 at the French Dental Association's international congress he demonstrated his system by creating a single posterior crown in less than an hour. Dr. Duret developed the Sopha CAD/CAM system later. In 1985 Dr. Mörmann developed the first commercial CAD/CAM system after consulting with Dr. Marco Brandestini, an electrical engineer. The device was called CEREC® system.

Also in the mid-1980s at the University of Minnesota, Dr. Rekow and her colleagues worked on a dental CAD/CAM system that used a 5-axis machine to mill the restorations.

Dr. Andersson in 1987 designed the Procera® system, he was trying hard by spark erosion to fabricate titanium copings. He also introduced CAD/CAM technology for processing composite veneered restorations.

Early CAD/CAM technology in dentistry permitted the fabrication of inlays, onlays, veneers, and single crowns.

Currently, CAD/CAM technology is able to provide fixed partial dentures, implant abutments, and surgical stents in dental implantology.(2) In addition the technology spread and developed to be able to fabricate complete dentures and removable partial denture frameworks.(3)

The CAD/CAM technology in dentistry is not only for restorations fabrication uses but also in orthodontics, it has been used to straighten teeth by using the Invisalign® technology.(4)

1.2 Dental CAD/CAM Systems overview

Dental CAD/CAM systems in general components are(4,5)

1. A scanner that's handheld, which captures the final tooth preparation and reflect its image to the monitor in a digital model.

2. Computer special software that receive the captured image from the scanner and allow the operator to design the final restoration design in a digital form and send its date to the milling machine.

3. Milling machine that mill the selected material block after the final design of the restoration is approved.

1.3 Advantages of CAD/CAM Systems in Dentistry

CAD/CAM technology in the dental field has strongly improved over the past recent decades. Many studies had evaluated the quality of CAD/CAM restorations.(6) The CAD/CAM technology was able to improve and provide the field of dentistry with numerous advantages of its restorations results over the traditional ones.(7,8)

These advantages involve time saving, ease to use, and better mechanical properties.

The elimination of multiple processing steps to manufacture the dental restoration reduced the cost and time.(9) In addition, some CAD/CAM machines has the ability to deliver the patient restoration in a single visit, thereby the need of temporization will be eliminated, that will save time and save material costs, and the patient will be more satisfied.(10)

The quality of the conventional restoration requires a high-level degree technician skill and proficiency. The ability to find such a level skill in a person is rear. However, the use of CAD/CAM system will allow the fabricated restorations with compatible, expected and reproducible result to be performed disregarding who is using the machine. For that, the required skill switch to CAD proficiency, that will allow mastering the job easier.(4,11,12)

The CAD/CAM material blocks from the same material kind is manufactured with equal quality standers, which provide the material with more consistencies. That will result in the elimination of porosity and will accomplish predictable conduct. On the other hand, conventional processing systems have a high chance of internal defects, which will vary the outcome of the restorations. Thus, they will lack the standardization processing.

The CAD/CAM material blocks are performed in a stander process, which gives it the preference over the conventional processing system materials.(8,13,14) The literature reviews for long-term clinical studies showed successful use of CAD/CAM dental restorations.(5,10,11,15–23)

1.4 Disadvantages of CAD/CAM Systems in dentistry

The main disadvantages of dental CAD/CAM systems nowadays are the initial cost of the machine, software, and training required for the operator to master the technology to be able to achieve the quality of the produced restorations, which will cost time and money. The dental office that plan to use CAD/CAM system should be able to overcome the initial financial issue and should also have a large flow of restoration production to get over the cost.(4,9)

According to some studies, the milling process for the CAD/CAM block material can affect some mechanical properties to the material. However, by the combination of polishing and overglazing the material will return the block material to its original strength.(12,13)

1.5 Ceramic Mechanics

At present the use of traditional porcelain-fused-to-metal prostheses has become less demanding for both patients and dentists, compared with the all-ceramic prostheses which are becoming more and more in use in the dental practice, patients have becoming more demanding regarding the dental restoration esthetics, and, durability. The main material of choice to reach the patient satisfaction was all-ceramic that challenged the scientists to develop new materials in the market every couple of years to reach the best material goal. However, all-ceramic prostheses are weaker, brittle and its fracture is commonly reported in the clinic especially in the posterior restorations quadrant where occlusal stress force is the most. To overcome this problem, there has been an accelerated development of new ceramics with different material compositions. Compared to the conventional porcelain which process under different firing steps, the recent manufacturing of ceramics have shifted to CAD/CAM Blocks which is productive and more standardized.(24–29)

By taking the first step to evaluate clinical behavior of biomaterials, which understands its mechanical properties.(30,31) There are three main properties that are commonly repeated regarding ceramic association with structural properties: (1) flexural strength, (2) fracture toughness, (3) capability with chemicals to assist crack growth.(31) Flexural strength is the ability of the material to bend before it brakes, which is one of the most important mechanical properties to evaluate clinical performance of the dental restorations, however, its not an inherent material property because its value depends on the material condition and the way the test was done.(32) Fracture toughness is the ability of the material containing a crack to resist fracture, which is also a major factor. One problem with ceramic materials in general and specifically dental ceramics is their fracture toughness is low. When fracture toughness is low that means the reliability of ceramic restoration in clinic will become low because the critical stress intensity level at which catastrophic failure occurs due to micro defect is defined by fracture toughness value. Therefore, the value of fracture toughness is as important as the flexural strength if not greater.(31,33)

1.6 Fracture Mechanics

Fractures have been a problem for decade, its is important that we save millions of money but more important to save human lives, that point got its interest during the World War II, when there was a widespread fractures to place in the welded Liberty Ships. At that time earnest investigation of brittle fracture started, the major problem was transition temperature from ductile to brittle, which affected the steel and caused the fractures. It was realized that understanding the fracture mechanics will lead to better structural constructions design by selecting materials that has the ability to resist fractures.(34)

1.6.1 Crack loading modes

Irwin introduced different three modes for crack loading. We will emphasize on mode I, which is the normal opening mode that occurs for most of the restorative materials in dentistry.(35)

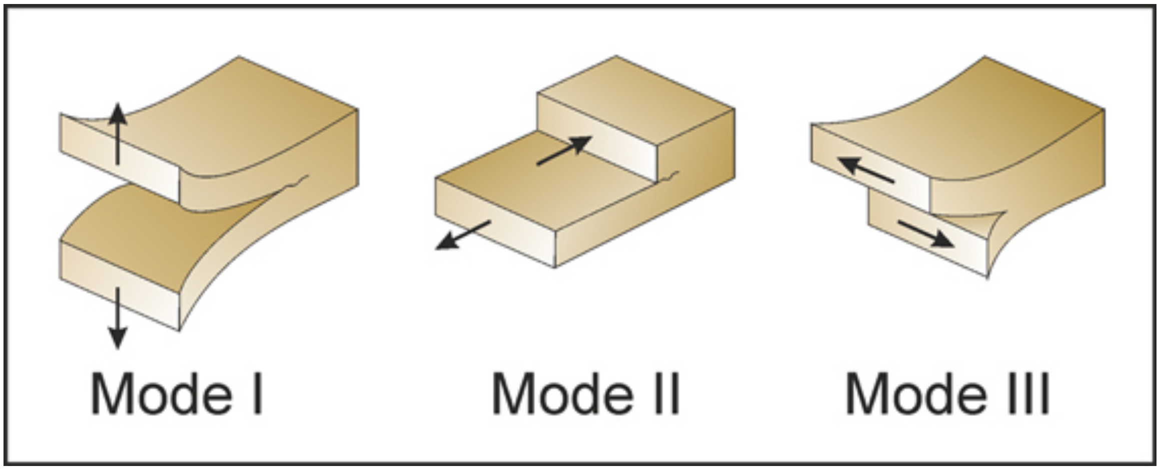

Figure 1: crack-loading modes. (36)

Mode I = opening mode (Tensile)

Mode II = sliding mode (In-plane shear)

Mode III = tearing mode (Out-of-plane shear)

1.6.2 Fracture Toughness

Fracture toughness in dentistry is one of the material properties used to characterize dental materials in vitro studies. It reflects the ability of the material containing a crack to resist fracture.(37)

When there is a material with an existing crack, there will be a stress area around the crack tip. The stress intensity caused is designated by "K", purely straight opening with tensile load is termed "mode I" opening. When stress increases and reaches critical point designated by "C" the crack will continue to become unstable and separate the material part into two pieces. Thus, fracture toughness is written "K_{IC}" (with units of $MPa \cdot m^{1/2}$) that represents the critical stress intensities for mode one opening and is used to compare different materials regardless the size of crack that exist.(31,38)

1.6.2.1 Fracture toughness testing methods for ceramics

Fracture toughness for ceramic materials had been difficult to achieve and had been much rare value for many years. Techniques such as double cantilever beam and double torsion was used to obtain a toughness value on an early structural ceramics. Crack length method was one of the thoughts for determining the value for ceramic toughness but for developing ceramic materials the specimen was not small and costly to prepare. Also the hard ability to form a sharp pre-crack after trial was a huge limitation to use the single-edged notched beam method technique. The development of Vickers-indentation method became more in use and preferred because it was simple to use, does not require a lot of specimen materials, and the results was acceptable. However, this method has a serious disadvantage. The main disadvantage is that the method depended on the simple and consistent crack pattern that developed from the indentation without considering ceramic materials. This is not the main issue as different groups of ceramics produce different crack forms in an indentation. In addition, the calculation of toughness value was determined by a lot of equations developed but the values calculated with other methods did not have any consistency in comparison between them, neither could these values support the information of other methods that could calculate the fracture toughness. At that time some literature articles recommended that Vickers-indentation methods in calculating the fracture toughness should be eliminated.(39–42)

In 1999, the American Society for Testing and Materials (ASTM) released standard test methods for determining the fracture toughness of ceramic materials (ASTM C1421).

The standard contains three methods for determining fracture toughness in different techniques— surface crack in flexure (SC), single-edge pre-cracked beam (PB), and chevron-notched beam (VB). Each method specimen requires having a sharp, well-defined pre-crack developed in it either during fracture (VB) or prior to fracture (SC and PB).

After (ASTM C1421) was published, the three methods were most popular in use and selected to determine the fracture toughness values for a lot of studies on ceramic materials. By reviewing the literature it shows that most of the studies depends on one method rather than selecting all of them. Although, selecting the right method for the test depends on the simplicity, rapidity, and familiarity to use.

All three methods subsumed in the (ASTM C1421) have undergone difficult and accurate technical evaluation. However, the ability to measure and determine the fracture toughness for all advanced ceramic by using the three methods only is very difficult to apply.

The (VB) method was strongly recommended over (SC and PB) methods because it is more controlled and dose not requires any post-test analysis. However, in this study we mainly used the PB method due to its applicability with the small size of the material specimens.

Furthermore, some studies applied different techniques for determining the fracture toughness of ceramic materials, for examples; novel fracture toughness test using a notchless triangular prism (NTP) specimen, laser notching for reliable fracture toughness, and compact tension test.

Given the very recent introduction and information of hybrid resin ceramic CAD/CAM technology and its materials, and the limited third-party research on them, it is important to evaluate their performance in order to validate or dispute their use. While no one property can be used to predict a material's clinical success or failure, several are important and play a role in the longevity and performance of a milled restoration. Fracture toughness and hardness fall into that category.(23)

Finally, it can be clinically relevant to verify the claims made by the manufacturers of these new ceramic materials. Based on manufacturers' documentation, these new materials have been tested for fracture toughness and they have compared to other commercially available CAD/CAM restorative materials, which they claim increases productivity.(43–46) However, as far as we know there is no study that tests the effect of cyclic loading for those materials when there is a crack or notch on their surface structure. Therefore, with the rapid development of the CAD/CAM ceramics in dentistry, there is a need to understand, predict and compare the fatigue behavior of those ceramics.

1.7 Statement of the problem

There is many CAD/CAM restorative materials have become available to the market and we don’t have complete understanding of their properties.

1.8 Objectives

The objectives of this study are as follows:

1. To evaluate the **fracture toughness** of the chairside CAD/CAM restorative materials included in the study.
2. To determine the **effect of cyclic loading after 100k and 200k** on the fracture toughness of the chairside CAD/CAM restorative materials tested.
3. To evaluate the **microhardness** of the chairside CAD/CAM restorative materials tested.
4. To determine the **effect of cyclic loading 100k and 200k** on the microhardness of the chairside CAD/CAM restorative materials.

Chapter 2. MATERIALS AND METHODS

2.1 Materials:

Five different CAD/CAM dental restorative materials with similar clinical indications were used in this in-vitro study.

The selected materials are as follows:

1- Lava™ Ultimate Restorative (3M ESPE, St. Paul, Minnesota)

2- IPS Empress® CAD (Ivoclar Vivadent AG, Schaan, Liechtenstein)

3- Enamic® (VITA Zahnfabrik, Bad Säckingen, Germany)

4- IPS e.max® CAD (Ivoclar Vivadent AG, Schaan, Liechtenstein)

5- CERASMART™ (GC Dental Products, Tokyo, Japan).

2.1.1 Lava™ Ultimate Restorative

Lava™ Ultimate Restorative (3M ESPE, St. Paul, Minnesota) is a fully cured composite resin material, according to the company they called it Resin Nano Ceramic (RNC) formulated using 80% by weight of zirconia nanoparticles (4 to 11 nm diameter), silica nanoparticles (20 nm diameter) and Nano clusters particles consist of bound aggregates of engineered nanoparticles (0.6 to 10 µm in size) that reinforce a resin matrix.(43) (Fig.2)

Figure 2: Lava™ Ultimate Restorative

2.1.2 IPS Empress® CAD

IPS Empress® CAD (Ivoclar Vivadent AG, Schaan, Liechtenstein) is a leucite reinforced glass ceramic of the SiO_2-Al_2O_3-K_2O materials system. The leucite crystals $KAlSi_2O_6$, which have been formed in a controlled process, endow the material with increased strength. The microstructure of the material is consisting of homogenously distributed leucite crystals (1-5 µm diameter). The crystal phase is 35-45% by volume.(44) (Fig.3)

Figure 3: IPS Empress® CAD

2.1.3 Enamic®

Enamic® (VITA Zahnfabrik, Bad Säckingen, Germany) is the first hybrid (infiltrated-ceramic network) material in market, which infiltrates the ceramic main structure with a monomer mixture of urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) and polymerized.

The composition of the ceramic part is (86 wt% / 75 vol%) and the composition of the polymer part is (14 wt% / 25 vol%).

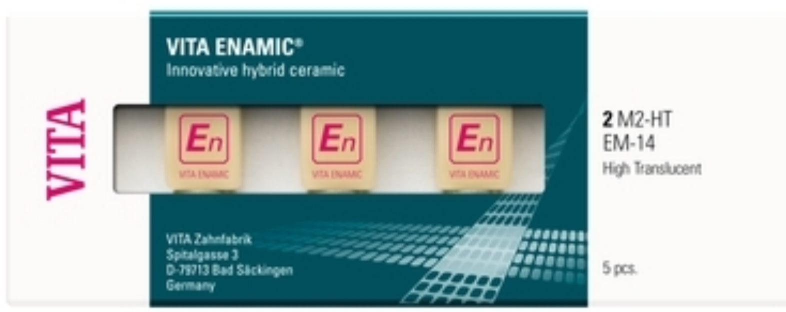

Figure 4: Enamic®

2.1.4 IPS e.max® CAD (Ivoclar Vivadent AG, Schaan, Liechtenstein)

Is a lithium diclicate glass-ceramic block material for CAD/CAM use. The blocks are partially crystallized, after milling the block, then crystallization process takes place approximately 25 minutes by firing the material up to 840 ^{o}C (1544 ^{o}F).

The final crystallization of the material consists of approximately 70% fine-grain of lithium dislicate crystals ($Li_2Si_2O_5$) embedded in a glassy matrix.(45)

Figure 5: IPS e.max® CAD

2.1.5 CERASMART™

CERASMART™ (GC Dental Products, Tokyo, Japan), The company claims that it is a flexible nano ceramic matrix structure, which has evenly distribution of ceramic particles. The company does still not reveal the exact microstructure and manufacturing process.

Figure 6: CERASMART™

2.2 Specimen Preparations and Testing Methods

2.2.1 Fracture toughness (K_{IC}) in static mode test using single edge pre crack beam (SEPB) method

A CAD/CAM block was sectioned into a grid of rectangular bars with a cross-section of 2x4 mm and a length of approximately 14 mm. The length of the bars varies with the dimensional constraints of the mill blocks.

The sectioning is performed using a 15LC diamond wafering blade with 0.5mm thickness mounted on an IsoMet™ 5000 Linear Precision Saw (Buehler, Lake Bluff, Illinois). The cuts are made at 800 rpm with 300 grams of load. The cutting area is constantly cooled by dual-nozzle water irrigation system.

Specimen dimensions are verified after sectioning using a micrometer (Model no. 293-715; Mitutoyo Corporation, Kanagawa, Japan).

Due to the varying dimensions between the different CAD/CAM blocks, the yield number of usable specimens varies slightly from material to material.

Single edge pre-crack beam (SEPB) method was followed to measure fracture toughness (K_{IC}), the notch was done by using a 15LC diamond wafering blade with 0.4mm thickness, blade mounted on an Isomet® 11-1180 Low Speed Saw (Buehler, Lake Bluff, Illinois). The cut are made at very slow speed with 150 grams of load. The cutting area is constantly cooled by water bath. The final notch depth was around 0.5-1 mm.

The specimen was polished using a Buehler EcoMet® 250 Grinder-Polisher (Buehler, Lake Bluff, Illinois). The sequence follows:

1-Burrs are removed on a 15-micron-grit diamond-polishing pad at 250 rpm with water irrigation.

2-Both sides of the specimen are run on a 15-micron-grit diamond-polishing pad at 250 rpm with water irrigation for 60 seconds for each side, and then thoroughly rinsed.

3-Finally, the two sides of the specimen are run on a Texmet® P polishing pad (Buehler, Lake Bluff, Illinois) with a 6-micron polycrystalline diamond suspension at 200 rpm for 90 seconds for each side, and then thoroughly rinsed.

A final check of specimen dimensions is made using a micrometer (Model no. 293- 715; Mitutoyo Corporation, Kanagawa, Japan). Any specimens with out-of-range dimensions are rejected.

The bars are also checked for chipping at the edges. Any bars with visible chipping are rejected and discarded as these imperfections can significantly influence flexural test results.(47–49)

The notch depth was measured by using a power microscope of Micromet® 2003 Microhardness Tester (Buehler, Lake Bluff, Illinois). The specimens are allowed to air dry for 24 hours before testing begins.

Three point bending test were carried out on the specimens using an Instron 5566A Universal Testing Frame (Instron, Norwood, Massachusetts) with 1 kN load cell.

The specimen is positioned on a fixture with a 10 mm support span in a way that the notch was placed at the bottom of the samples and is centered. Under the loading apparatus and aligned to be perpendicular to it.

The test is run with a crosshead speed of 0.5mm/min, controlled using BlueHill 3 software (Instron, Norwood, Massachusetts). Each specimen is loaded with the force to failure.

The controlling software calculates the maximum load (N) and maximum extension (mm). Fractured pieces of the specimen are collected and stored in a sealed plastic sleeve for future uses.

Figure 7 illustrates a typical three-point flexural test set-up (50) where:

- F is the applied load.
- L is the support span.
- b is the width of the rectangular specimen.
- d is the height of the rectangular specimen.
- a is the notch depth.

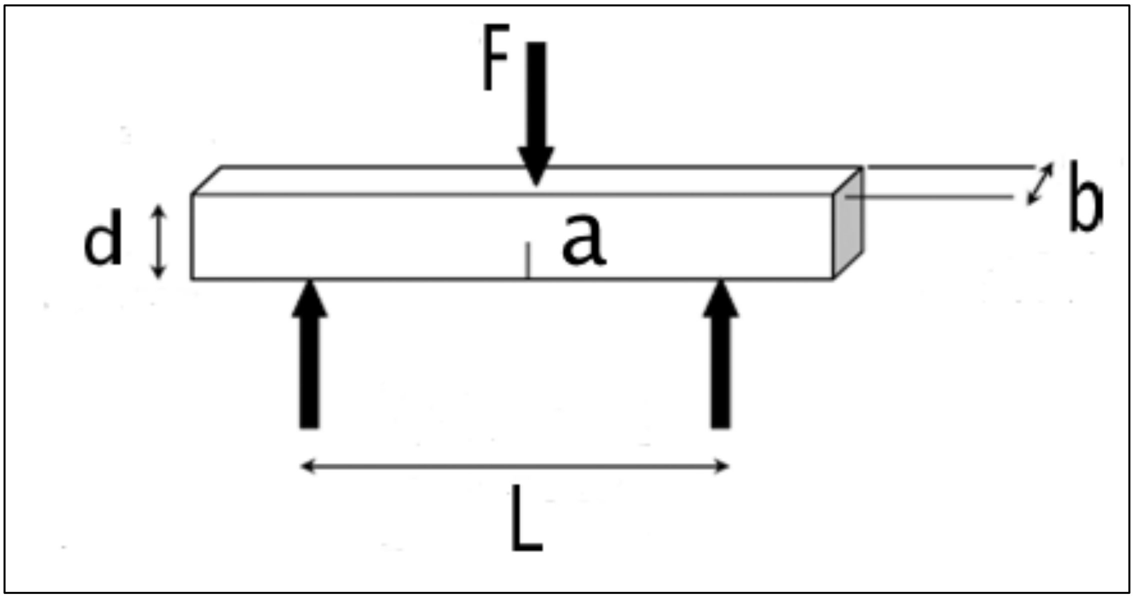

Figure 7: Three-point flexural test set-up

The data collected by a bending test, combined with the post failure specimen measurements, permit the calculation of the values of fracture toughness ($MPa \cdot m^{1/2}$) for all specimens according to the equation of fracture toughness as shown below:

$$K_{1C} = Y \sigma \sqrt{a}$$

Where:

- Y is the dimensionless stress intensity shape factor.
- σ is the fracture strength.
- a is the notch depth.

The results of the calculations are expressed as means and standard deviations for each material tested. Differences in fracture toughness are analyzed by means of a oneway analysis of variance (ANOVA) with **α**=0.05.

Post-hoc testing is performed using the Tukey HSD (Honestly Significant Difference) test with **α** =0.05 for comparison of the means between the different materials. All statistical analysis are performed using the JMP Pro 12.0 statistics software package.

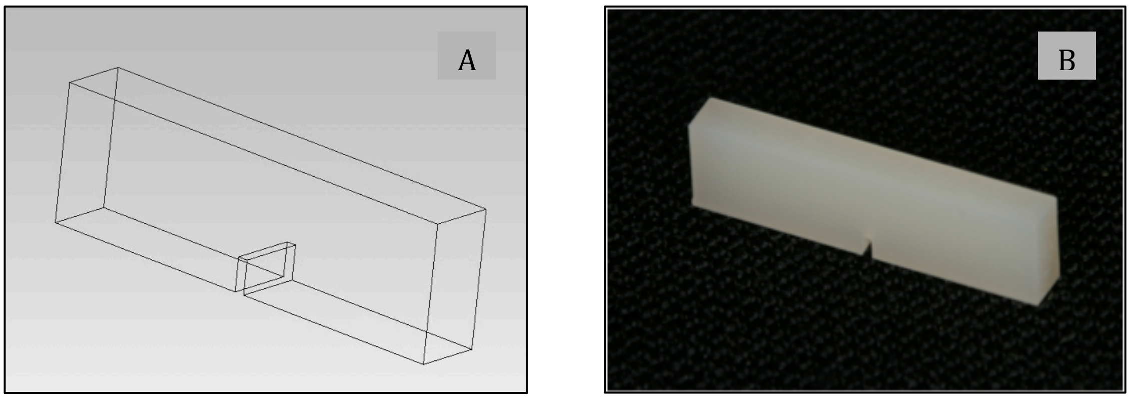

Figure 8: Images showing the specimen shape used in the study. (A) 3D drawing. (B) Actual specimen.

2.2.2 Fracture toughness (K_{1C}) after 100k and 200k cyclic loading test using single edge pre crack beam (SEPB) method

A group of rectangular specimens with notch per material were prepared and polished as mentioned previously, then these were subjected to 100k and 200k loading cycles at 1 Hz in water baths under a pneumatically driven fatigue tester using a maximum load of 55 N The cyclic loaded specimens were then subjected to the same three-point bending test.

2.2.3 Microhardness test

Fifteen specimens were prepared by emending each three specimens in epoxy material in a cylindrical shape box and wait 24 hours for it to set. Then the cylindrical blocks were polished until it was clear to see through it. Each material group was included in static mode, after 100k cyclic loading, and after 200k cyclic loading.

The microhardness tests were performed according to ISO 6507 standard with Vickers indenter. The Vickers indentation process was done and measuring the diagonal lengths of the indentations on the material surface with Micromet® 2003 Microhardness Tester (Buehler, Lake Bluff, Illinois). The applied load was adjusted to 300 g for Enamic®, CERASMART™ and Lava™ Ultimate Restorative, while it was adjusted to 500 g for both IPS e.max® CAD and IPS Empress® CAD. The dwelling time was 15 seconds. Ten measurements were made on each sample and the mean was calculated.

2.2.4 Fracture toughness (K_{1C}) test using indentation fracture (IF) method

The indentation fracture (IF) method was used to measure the fracture toughness for both IPS e.max® CAD and IPS Empress® CAD materials specimens.

After the Vickers indentation process was done in the microhardness test, the specimens micro fracture crack length was measured under the SEM by using Quartz PCI software. The data collected was used to calculate the fracture toughness ($MPa \cdot m^{1/2}$) for all specimens according to the equation as shown below:

$$K_{1c} = B\left(\frac{E}{H}\right)^{1/2}\left(\frac{W}{c^{3/2}}\right)$$

Where:

B is an empirical constant, 0.016

W is the load in Newton.

$c^{3/2}$ is the crack lengths from the center of the indent to the crack tip in meters.

E is the Young's modulus in GPa.

H is the Vickers hardness in GPa.

The fracture toughness (K_{1C}) by using this method was calculated and then compared with the fracture toughness (K_{1C}) by using single edge pre crack beam (SEPB) method.

Chapter 3. RESULTS

3.1 Fracture toughness (K_{1C}) by single edge pre crack beam (SEPB) method

The fracture toughness of five CAD/CAM dental restorative materials determined by using single edge pre crack beam (SEPB) method was compared by material treatments and material types by using oneway ANOVA statistical analysis.

3.1.2 Comparison of fracture toughness (K_{1C}) by material treatments

The fracture toughness of five CAD/CAM dental restorative materials was compared by material treatments (in static mode treatment, after 100k cyclic loading treatment, and after 200k cyclic loading treatment) by using oneway ANOVA and results follows.

3.1.2.1 Fracture toughness (K_{1C}) in static mode treatment

The mean fracture toughness (MPa·m$^{1/2}$), standard deviation and coefficient of variance for each material were calculated.

Table 1: Mean fracture toughness (MPa·m$^{1/2}$), standard deviation and coefficient of variance for the materials tested in static mode treatment.

Material	N	Mean	SD	CV
e.max CAD	10	3.20	0.21	6.50
Empress CAD	10	1.95	0.19	9.79
Cerasmart	10	1.65	0.61	36.99
Enamic	10	1.92	0.29	14.94
Lava Ult	10	2.00	0.60	30.06

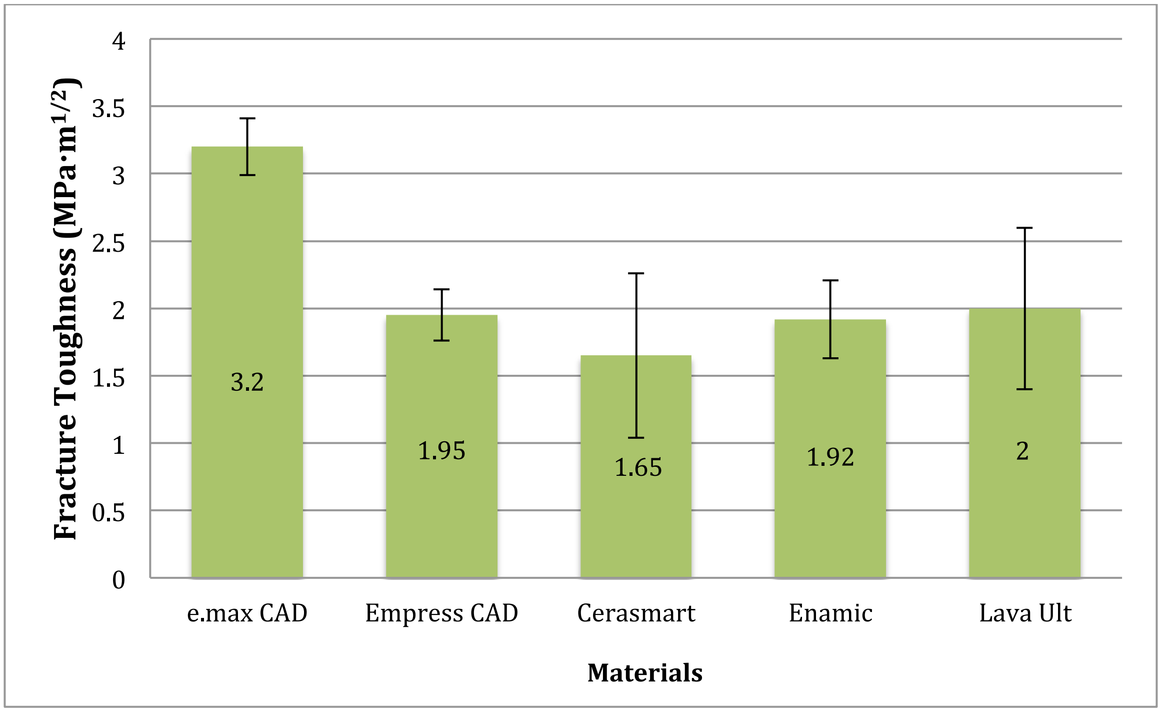

Figure 9: Mean fracture toughness (MPa·m$^{1/2}$) by SEPB of tested materials in static mode treatment.

The fracture toughness of the material tested in static mode ranged from 3.2 ± 0.21 MPa·m$^{1/2}$ for e.max CAD, to 1.65 ± 0.61 MPa·m$^{1/2}$ for Cerasmart. Lava Ult had a fracture toughness of 2.00 ± 0.60 MPa·m$^{1/2}$, followed by Empress CAD 1.95 ± 0.19 MPa·m$^{1/2}$, and Enamic 1.92 ± 0.29 MPa·m$^{1/2}$ respectively.

In order to determine which specific group mean different, an order differences report and connecting letters report are listed.

Table 2: Ordered differences report for all materials tested in static mode.

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
e.maxCAD	Cerasmart	1.547400	0.189285 2	1.00956	2.085245	<.0001*
e.maxCAD	Enamic	1.280400	0.189285 2	0.74256	1.818245	<.0001*
e.maxCAD	EmpressCAD	1.250900	0.189285 2	0.71306	1.788745	<.0001*
e.maxCAD	LavaUlt	1.201100	0.189285 2	0.66326	1.738945	<.0001*
LavaUlt	Cerasmart	0.346300	0.189285 2	-0.19154	0.884145	0.3699
Empress	Cerasmart	0.296500	0.189285 2	-0.24134	0.834345	0.5261
Enamic	Cerasmart	0.267000	0.189285 2	-0.27084	0.804845	0.6242
LavaUlt	Enamic	0.079300	0.189285 2	-0.45854	0.617145	0.9933
LavaUlt	EmpressCAD	0.049800	0.189285 2	-0.48804	0.587645	0.9989
EmpressCAD	Enamic	0.029500	0.189285 2	-0.50834	0.567345	0.9999

Table 3: Connecting letters report for all materials tested in static mode.

Level	Letter	Mean
e.max CAD	A	3.1990
LavaUlt	B	1.9979
Empress CAD	B	1.9481
Enamic	B	1.9186
Cerasmart	B	1.6516

3.1.2.2 Fracture toughness (K_{1C}) after 100k cycles loading treatment

The mean fracture toughness (MPa·$m^{1/2}$), standard deviation, and coefficient of variance for each material were calculated.

Table 4: Mean fracture toughness (MPa·$m^{1/2}$), standard deviation and coefficient of variance for the materials tested after 100k cyclic loading treatments.

Material	N	Mean	SD	CV
e.max CAD	10	4.02	1.00	24.86
Empress CAD	10	1.94	0.34	17.49
Cerasmart	10	3.06	0.47	15.25
Enamic	10	2.01	0.19	9.46
Lava Ult	10	2.55	0.33	12.85

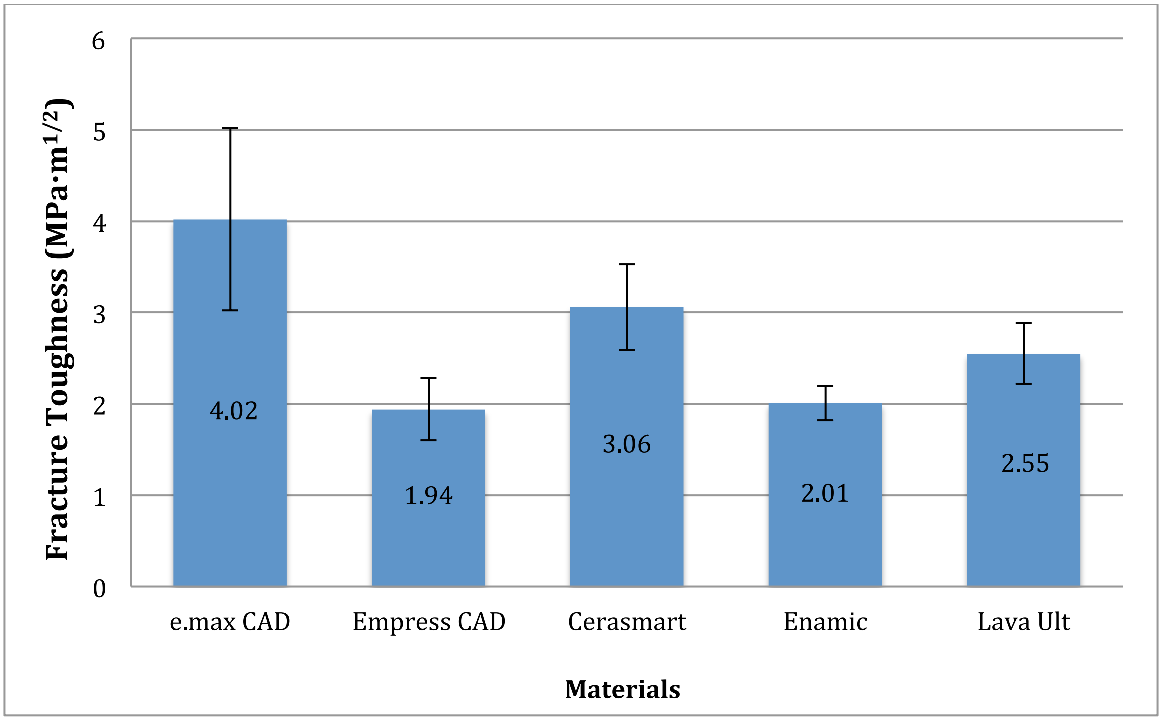

Figure 10: Mean fracture toughness (MPa·m$^{1/2}$) by SEPB and standard deviation of tested materials after 100k cyclic loading treatment.

The fracture toughness of the material tested after 100k cyclic loads ranged from 4.02 ± 1.00 MPa·m$^{1/2}$ for e.max CAD, to 1.94 ± 0.34 MPa·m$^{1/2}$ for Empress CAD. Cerasmart had a fracture toughness of 3.06 ± 0.47 MPa·m$^{1/2}$, followed by Lava Ult 2.55 ± 0.33 MPa·m$^{1/2}$, and Enamic 2.01 ± 0.19 MPa·m$^{1/2}$ respectively.

In order to determine which specific group means are different, an order differences report and connecting letters report are listed below.

Table 5: Ordered differences report for all materials tested after 100k cyclic loading.

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
e.maxCAD	EmpressCAD	2.076140	0.2366353	1.40548	2.746804	<.0001*
e.maxCAD	Enamic	1.901492	0.2225649	1.27071	2.532279	<.0001*
e.maxCAD	LavaUlt	1.468830	0.2366353	0.79817	2.139494	<.0001*
Cerasmart	EmpressCAD	1.123440	0.2366353	0.45278	1.794104	0.0002*
e.maxCAD	Cerasmart	0.952700	0.2366353	0.28204	1.623364	0.0018*
Cerasmart	Enamic	0.948792	0.2225649	0.31801	1.579579	0.0009*
LavaUlt	EmpressCAD	0.607310	0.2366353	-0.06335	1.277974	0.0931
Cerasmart	LavaUlt	0.516130	0.2366353	-0.15453	1.186794	0.2043
LavaUlt	Enamic	0.432662	0.2225649	-0.19812	1.063449	0.3088
Enamic	EmpressCAD	0.174648	0.2225649	-0.45614	0.805434	0.9338

Table 6: Connecting letters report for all materials tested after 100k cyclic loading.

Level	Letter			Mean
e.max CAD	A			4.0158
Cerasmart		B		3.0631
LavaUlt		B	C	2.5469
Enamic			C	2.1143
Empress CAD			C	1.9396

3.1.2.3 Fracture toughness (K_{1C}) after 200k cycles loading treatment

The mean fracture toughness (MPa·$m^{1/2}$), standard deviation, and coefficient of variance for each material were calculated.

Table 7: Mean fracture toughness (MPa·$m^{1/2}$), standard deviation and coefficient of variance for the materials tested after 200k cyclic loading treatments.

Material	N	Mean	SD	CV
e.max CAD	14	2.68	0.37	13.71
Empress CAD	10	1.72	0.42	24.39
Cerasmart	10	3.14	0.39	12.37
Enamic	10	2.01	0.09	4.23
Lava Ult	10	2.83	0.24	8.59

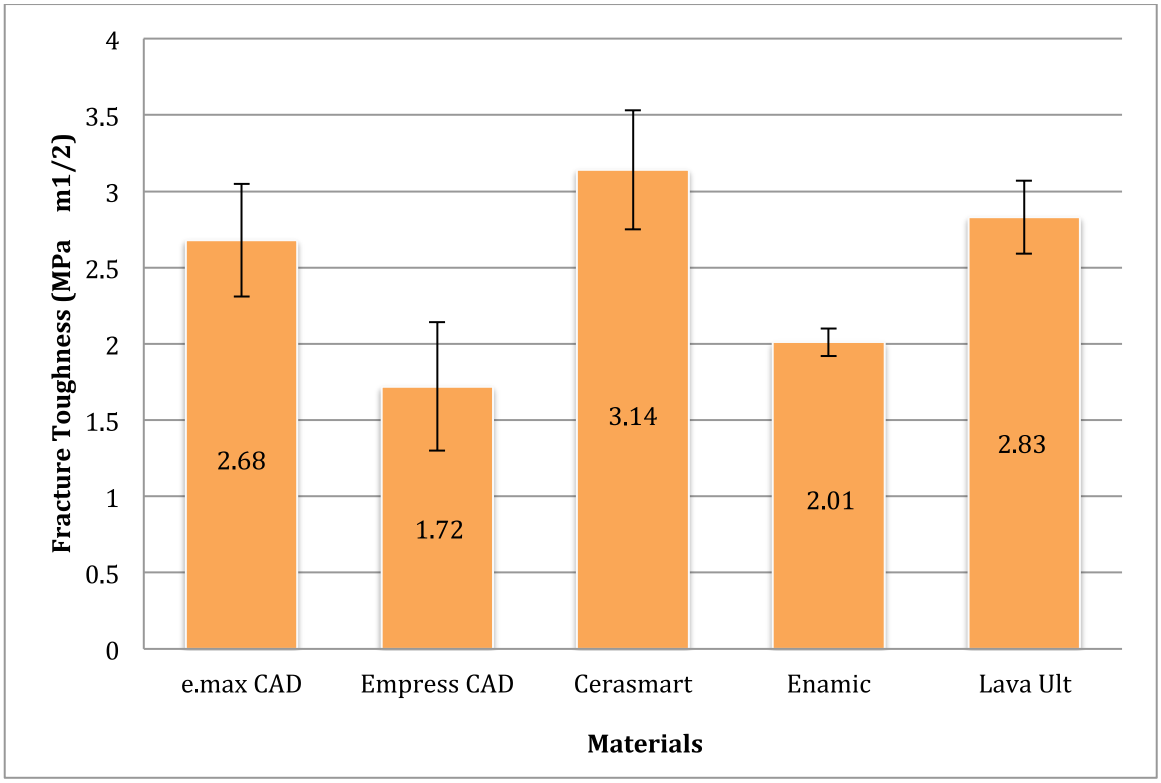

Figure 11: Mean fracture toughness (MPa·m$^{1/2}$) by SEPB and standard deviation of tested materials after 200k cyclic loading treatment.

The fracture toughness of the material tested after 200k cyclic loads ranged from 3.14 ± 0.39 MPa·m$^{1/2}$ for Cerasamrt, to 1.72 ± 0.42 MPa·m$^{1/2}$ for Empress CAD. Lava Ult had a fracture toughness of 2.83 ± 0.24 MPa·m$^{1/2}$, followed by e.max CAD 2.68 ± 0.37 MPa·m$^{1/2}$, and Enamic 2.01 ± 0.09 MPa·m$^{1/2}$ respectively.

In order to determine which specific group means are different, an order differences report and connecting letters report are listed.

Table 8: Ordered differences report for all materials tested after 200k cyclic loading.

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
Cerasmart	EmpressCAD	1.424950	0.1468227	1.00916	1.840744	<.0001*
Cerasmart	Enamic	1.128200	0.1468227	0.71241	1.543994	<.0001*
LavaUlt	EmpressCAD	1.115110	0.1468227	0.69932	1.530904	<.0001*
e.maxCAD	EmpressCAD	0.961707	0.1359314	0.57676	1.346657	<.0001*
LavaUlt	Enamic	0.818360	0.1468227	0.40257	1.234154	<.0001*
e.maxCAD	Enamic	0.664957	0.1359314	0.28001	1.049907	0.0001*
Cerasmart	e.max CAD	0.463243	0.1359314	0.07829	0.848193	0.0110*
Cerasmart	LavaUlt	0.309840	0.1468227	-0.10595	0.725634	0.2322
Enamic	EmpressCAD	0.296750	0.1468227	-0.11904	0.712544	0.2714
LavaUlt	e.maxCAD	0.153403	0.1359314	-0.23155	0.538353	0.7907

Table 9: Connecting letters report for all materials tested after 200k cyclic loading.

Level	Letter	Mean
Cerasmart	A	3.1406
LavaUlt	A B	2.8307
e.max CAD	B	2.6773
Enamic	C	2.0124
Empress CAD	C	1.7156

3.1.3 Comparison of fracture toughness (K_{1C}) in static mode and after fatigue by material types

The fracture toughness of five CAD/CAM dental restorative materials determined by using SEPB method was compared by material type by using oneway ANOVA which follows in the next five sections.

3.1.3.1 Lava™ Ultimate Restorative

The fracture toughness in static mode, after 100k cyclic loading, and after 200k cyclic loading for Lava™ Ultimate Restorative material were compared by using oneway ANOVA statistical analysis that follows.

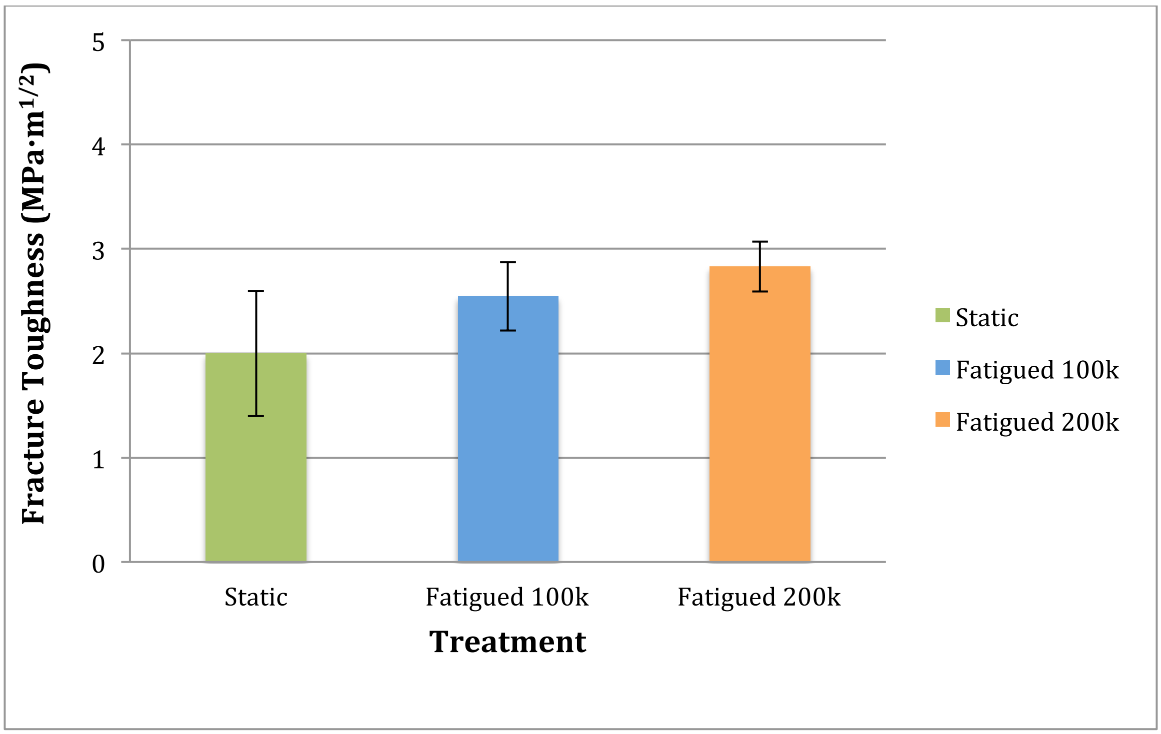

Figure 12: Mean fracture toughness (MPa·m$^{1/2}$) of different treatments for Lava™ Ultimate Restorative material.

In order to determine which specific group means are different, a comparisons for all pairs using *post hoc* Tukey-Kramer HSD test, with alpha=0.05. The results of the test are shown in Table 15.

Table 10: Tukey HSD test results of fracture toughness ($MPa \cdot m^{1/2}$) parameter for Lava™ Ultimate Restorative material.

Abs(Dif)-HSD	Fatigued 200k	Fatigued 100k	Static
Fatigued 200k	-0.46469	-0.18090	0.36817
Fatigued 100k	-0.18090	-0.46469	0.08438
Static	0.36817	0.08438	-0.46469

Table 11: Connecting letters report for Lava™ Ultimate Restorative material.

Level	Letter		Mean
Static		B	1.9979
Fatigued 100k	A		2.5469
Fatigued 200k	A		2.8307

3.1.3.2 IPS Empress® CAD

The fracture toughness in static mode, after 100k cyclic loading, and after 200k cyclic loading for IPS Empress® CAD material were compared by using oneway ANOVA statistical analysis that follows.

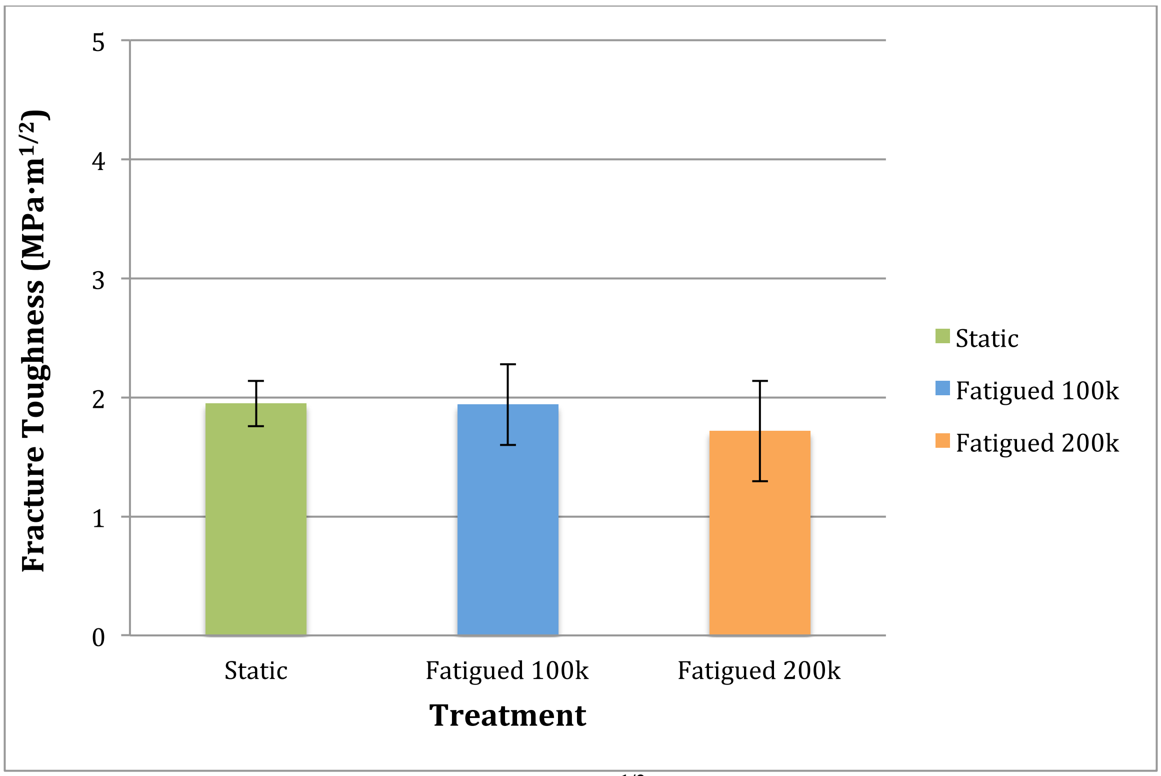

Figure 13: Mean fracture toughness (MPa·m$^{1/2}$) of different treatments for IPS Empress® CAD material.

In order to determine which specific group means are different, a comparisons for all pairs using *post hoc* Tukey-Kramer HSD test, with alpha=0.05. The results of the test are shown in Table 17.

Table 12: Tukey HSD test results of fracture toughness (MPa·$m^{1/2}$) parameter for IPS Empress® CAD material.

Abs(Dif)-HSD	Static	Fatigued 100k	Fatigued 200k
Static	-0.36578	-0.35734	-0.13333
Fatigued 100k	-0.35734	-0.36578	-0.14177
Fatigued 200k	-0.13333	-0.14177	-0.36578

Table 13: Connecting letters report for IPS Empress® CAD material.

Level	Letter	Mean
Static	A	1.9481
Fatigued 100k	A	1.9396
Fatigued 200k	A	1.7156

3.1.3.3 Enamic®

The fracture toughness in static mode, after 100k cyclic loading, and after 200k cyclic loading for Enamic® material were compared by using oneway ANOVA statistical analysis that follows.

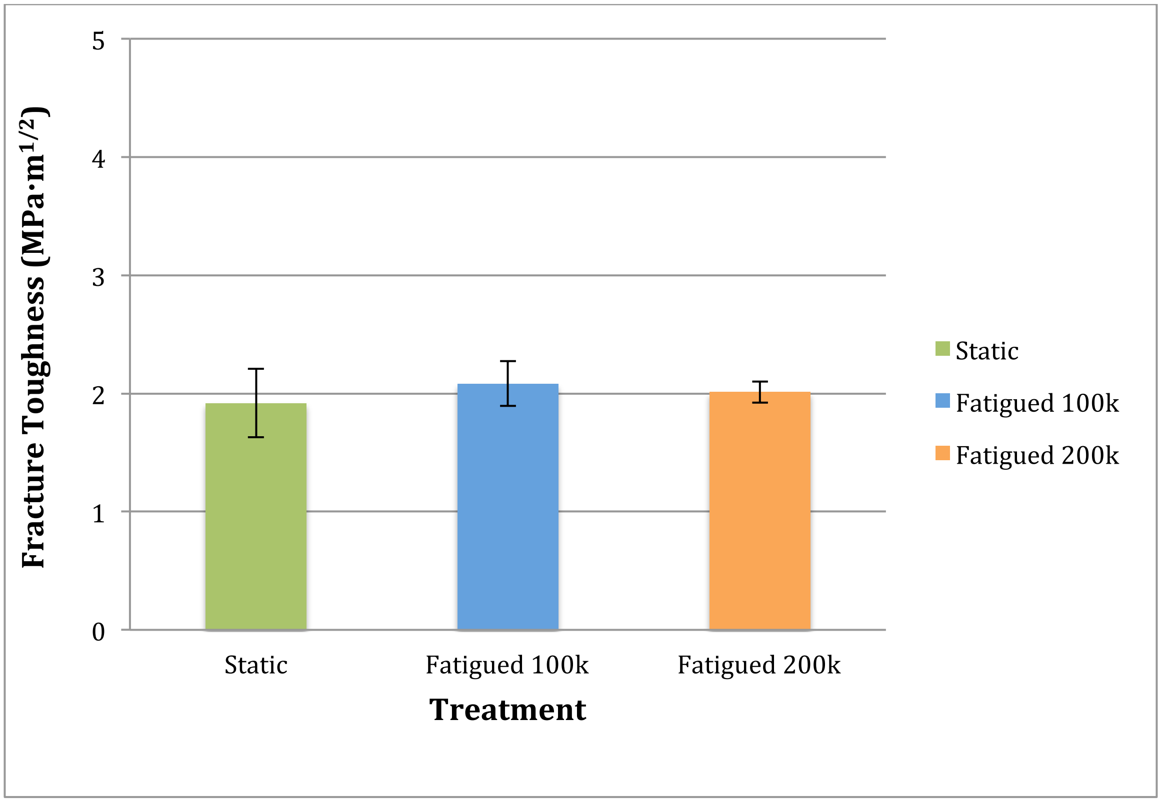

Figure 14: Mean fracture toughness (MPa·m$^{1/2}$) of different treatments for Enamic® material.

In order to determine which specific group means are different, a comparisons for all pairs using *post hoc* Tukey-Kramer HSD test, with alpha=0.05. The results of the test are shown in Table 19.

Table 14: Tukey HSD test results of fracture toughness ($MPa \cdot m^{1/2}$) parameter for Enamic® material.

Abs(Dif)-HSD	Fatigued 100k	Fatigued 200k	Static
Fatigued 100k	-0.20223	-0.11496	-0.02116
Fatigued 200k	-0.11496	-0.23057	-0.13677
Static	-0.02116	-0.13677	-0.23057

Table 15: Connecting letters report for Enamic® material.

Level	Letter	Mean
Static	A	1.9186
Fatigued 100k	A	2.1143
Fatigued 200k	A	2.0124

3.1.3.4 IPS e.max® CAD

The fracture toughness in static mode, after 100k cyclic loading, and after 200k cyclic loading for IPS e.max® CAD material were compared by using oneway ANOVA statistical analysis that follows.

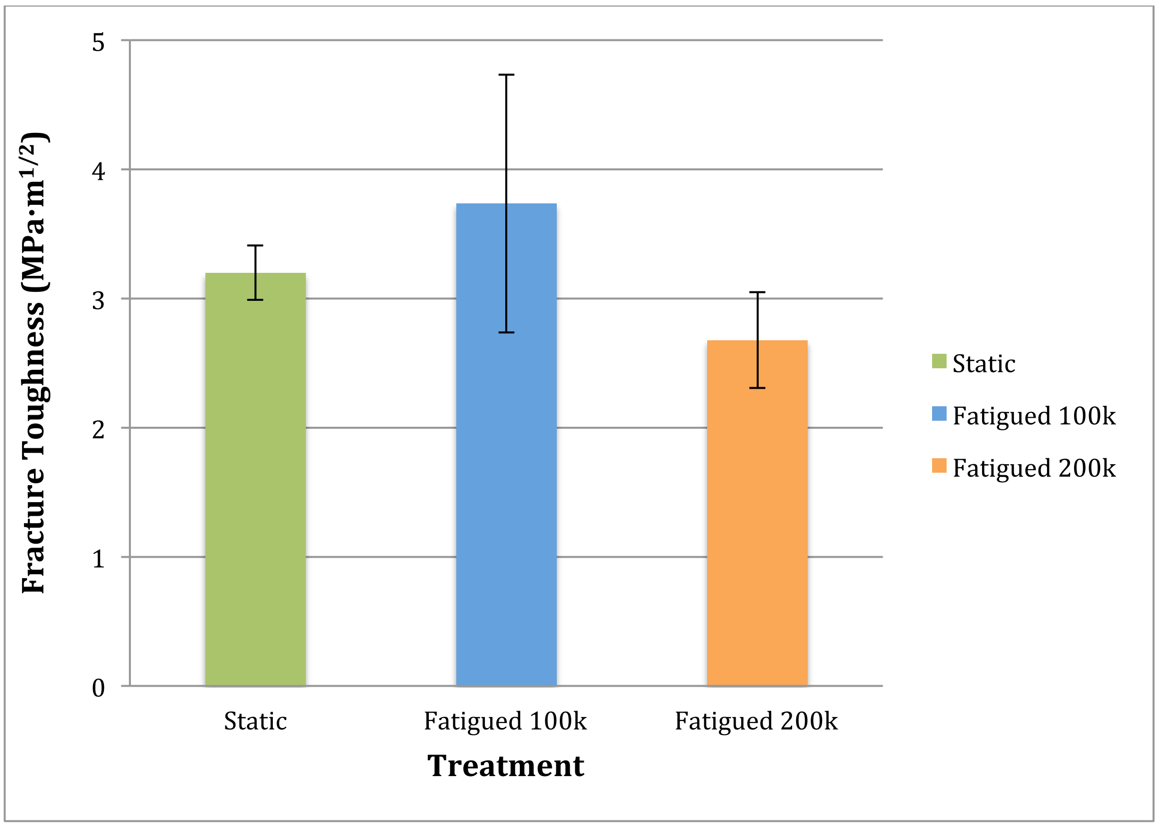

Figure 15: M Mean fracture toughness (MPa·m$^{1/2}$) of different treatments for IPS e.max® CAD material.

In order to determine which specific group means are different, a comparisons for all pairs using *post hoc* Tukey-Kramer HSD test, with alpha=0.05. The results of the test are shown in Table 21.

Table 16: Tukey HSD test results of fracture toughness ($MPa \cdot m^{1/2}$) parameter for IPS e.max® CAD material.

Abs(Dif)-HSD	Fatigued 100k	Static	Fatigued 200k
Fatigued 100k	-0.65887	0.15793	0.72845
Static	0.15793	-0.65887	-0.08835
Fatigued 200k	0.72845	-0.08835	-0.55684

Table 17: Connecting letters report for IPS e.max® CAD material.

Level	Letter	Mean
Static	B	3.1990
Fatigued 100k	A	4.0158
Fatigued 200k	B	2.6773

3.1.3.5 CERASMART™

The fracture toughness of static mode, after 100k cyclic loading, and after 200k cyclic loading for CERASMART™ material were compared by using oneway ANOVA statistical analysis that follows.

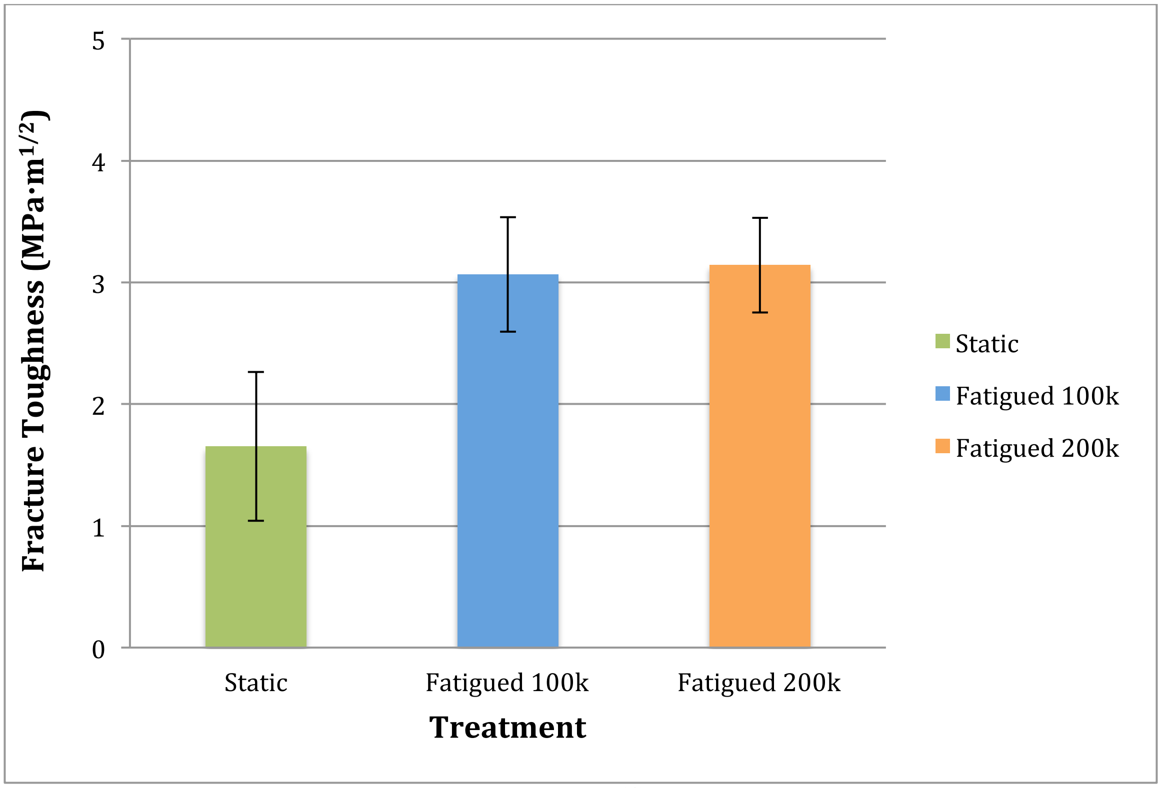

Figure 16: Mean fracture toughness (MPa·$m^{1/2}$) of different treatments for CERASMART™ material.

In order to determine which specific group means are different, a comparisons for all pairs using *post hoc* Tukey-Kramer HSD test, with alpha=0.05. The results of the test are shown in Table 23.

Table 18: Tukey HSD test results of fracture toughness ($MPa\cdot m^{1/2}$) parameter for CERASMART™ material.

Abs(Dif)-HSD	Fatigued 200k	Fatigued 100k	Static
Fatigued 200k	-0.55161	-0.47411	0.93739
Fatigued 100k	-0.47411	-0.55161	0.85989
Static	0.93739	0.85989	-0.55161

Table 19: Connecting letters report for CERASMART™ material.

Level	Letter	Mean
Static	B	1.6516
Fatigued 200k	A	3.1406
Fatigued 100k	A	3.0631

3.1.4 Interaction of materials and treatments

In order to determine which specific interaction between materials and treatments group means are different, a comparisons for all pairs using connecting letter report that follows.

Level	Letter	Least Sq Mean
emax CAD,Fatigued 100k	A	4.0158
emax CAD,Static	B	3.1990
CeraSmart,Fatigued 200k	B	3.1406
CeraSmart,Fatigued 100k	B	3.0631
LavaUlt,Fatigued 200k	B	2.8307
emax CAD,Fatigued 200k	B C	2.6773
LavaUlt,Fatigued 100k	B C D	2.5469
Enamic,Fatigued 100k	C D E	2.1143
Enamic,Fatigued 200k	D E	2.0124
LavaUlt,Static	D E	1.9979
Empress,Static	D E	1.9481
Empress,Fatigued 100k	D E	1.9396
Enamic,Static	D E	1.9186
Empress,Fatigued 200k	E	1.7156
CeraSmart,Static	E	1.6516

3.2 Microhardness test

The Vickers hardness number (VHN) of the five CAD/CAM dental restorative materials that were tested was determined by using Micromet® 2003 Microhardness Tester (Buehler, Lake Bluff, Illinois). The mean, standard deviation, and coefficient of variance for each material were calculated.

Table 20: Mean Vickers hardness number (kg/mm^2), standard deviation and coefficient of variance for the materials tested.

Material	Treatment	N	Mean	SD	CV
Cerasmart	Static	20	68.69	2.27	3.31
	100k	20	70.35	2.28	3.23
	200k	20	68.95	3.12	4.53
e.max CAD	Static	16	515.59	11.76	2.28
	100k	20	506.26	13.67	2.70
	200k	20	488.23	12.39	2.54
Empress CAD	Static	20	457.61	24.87	5.44
	100k	20	479.93	19.12	3.98
	200k	20	467.51	22.94	4.91
Enamic	Static	16	196.61	12.94	6.58
	100k	18	215.97	18.95	8.77
	200k	16	211.65	33.47	15.81
LavaUlt	Static	20	107.08	2.43	2.27
	100k	20	113.12	7.90	6.99
	200k	20	112.57	9.97	8.85

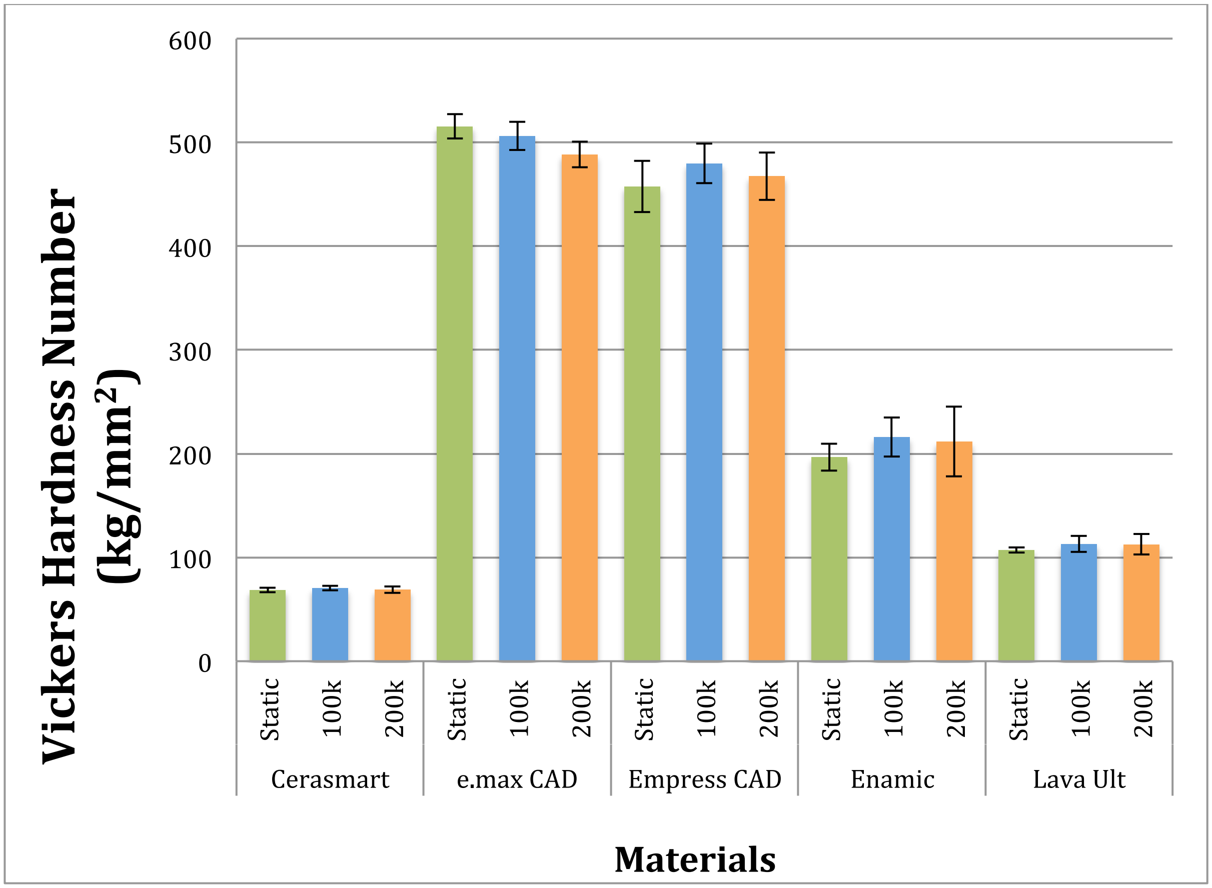

Figure 17: Mean Vickers hardness number (kg/mm^2) and standard deviation for the materials tested.

The Vickers hardness number (VHN) of the material tested in static mode ranged from 515.59 ± 11.76 kg/mm^2 for e.max CAD, to 68.69 ± 2.27 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 457.61± 24.87 kg/mm^2, followed by Enamic 196.61 ± 12.94 kg/mm^2, and Lava Ult 107.08 ± 2.43 kg/mm^2 respectively.

The Vickers hardness number (VHN) of the material tested after 100k cyclic loads ranged from 506.26 ± 13.67 kg/mm^2 for e.max CAD, to 70.35 ± 2.28 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 479.93 ± 19.12 kg/mm^2, followed by Enamic 215.97 ± 18.95 kg/mm^2, and Lava Ult 113.12 ± 7.90 kg/mm^2 respectively.

The Vickers hardness number (VHN) of the material tested after 200k cyclic loads ranged from 488.23 ± 12.39 kg/mm^2 for e.max CAD, to 68.95 ± 3.12 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 467.51± 22.94 kg/mm^2, followed by Enamic 211.65 ± 33.47 kg/mm^2, and Lava Ult 112.57 ± 9.97 kg/mm^2 respectively.

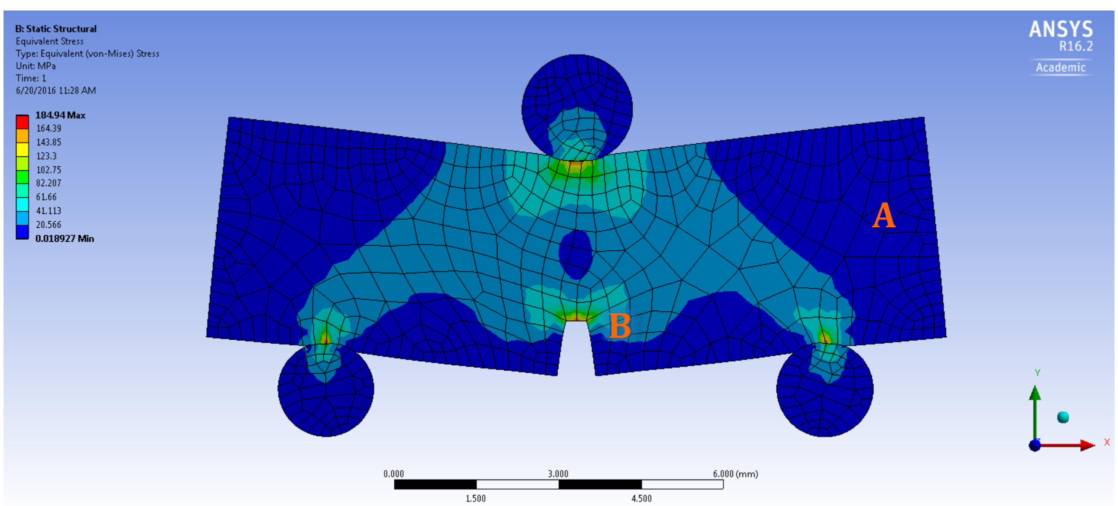

Figure 18: Stress distribution from finite elemental analysis of singe edge notch beam showing two different stress areas, where A=low stress area, B=high stress area.

The Vickers microhardness test was done in two areas for each specimen, the first area that we named area A, low stress area, that is the area away from the notch area and the second area we named area B, high stress area, that is at the notch tip. (Fig.18)

3.2.1 Comparison of microhardness test of materials by stress area

The Vickers hardness number (VHN) of the five CAD/CAM dental restorative materials was determined, and then a comparison was done by stress area by using different statistical analysis that follows.

3.2.1.1 Lava™ Ultimate Restorative

Using different statistical analysis that follows compared the Vickers hardness number in low stress area and in high stress area.

Table 21: Mean Vickers hardness number (kg/mm^2) by stress area for Lava™ Ultimate Restorative.

Level	N	Mean
A= low stress	30	107.593
B=high stress	30	114.247

Table 22: Tukey HSD test results of Vickers hardness number (kg/mm^2) by stress area for Lava™ Ultimate Restorative.

Abs(Dif)-HSD	B=high stress	A= low stress
B=high stress	-3.6980	2.9553
A= low stress	2.9553	-3.6980

3.2.1.2 IPS Empress® CAD

Using different statistical analysis that follows compared the Vickers hardness number in low stress area and in high stress area.

Table 23: Mean Vickers hardness number (kg/mm^2) by stress area for IPS Empress® CAD.

Level	N	Mean
A= low stress	30	464.867
B=high stress	30	471.830

Table 24: Tukey HSD test results of Vickers hardness number (kg/mm^2) by stress area for IPS Empress® CAD.

Abs(Dif)-HSD	B=high stress	A= low stress
B=high stress	-12.325	-5.362
A= low stress	-5.362	-12.325

3.2.1.3 Enamic®

Using different statistical analysis that follows compared the Vickers hardness number in low stress area and in high stress area.

Table 25: Mean Vickers hardness number (kg/mm^2) by stress area for Enamic®.

Level	N	Mean
A= low stress	25	215.872
B=high stress	25	200.916

Table 26: Tukey HSD test results of Vickers hardness number (kg/mm^2) by stress area for Enamic®.

Abs(Dif)-HSD	A= low stress	B=high stress
A= low stress	-13.248	1.708
B=high stress	1.708	-13.248

3.2.1.4 IPS e.max® CAD

Using different statistical analysis that follows compared the Vickers hardness number in low stress area and in high stress area.

Table 27: Mean Vickers hardness number (kg/mm^2) by stress area for IPS e.max® CAD.

Level	N	Mean
A= low stress	28	499.168
B=high stress	28	505.800

Table 28: Tukey HSD test results of Vickers hardness number (kg/mm^2) by stress area for IPS e.max® CAD.

Abs(Dif)-HSD	B=high stress	A= low stress
B=high stress	-8.9377	-2.3056
A= low stress	-2.3056	-8.9377

3.2.1.5 CERASMART™

Using different statistical analysis that follows compared the Vickers hardness number in low stress area and in high stress area.

Table 29: Mean Vickers hardness number (kg/mm^2) by stress area for CERASMART™.

Level	N	Mean
A= low stress	30	68.6400
B=high stress	30	70.0133

Table 30: Tukey HSD test results of Vickers hardness number (kg/mm^2) by area for CERASMART™.

Abs(Dif)-HSD	B=high stress	A= low stress
B=high stress	-1.3319	0.0414
A= low stress	0.0414	-1.3319

3.2.1.6 VHN comparison in low stress area (A zone)

The mean Vickers hardness numbers (VHN) of the five CAD/CAM dental restorative materials in low stress area are shown in Table 41.

Table 31: Mean Vickers hardness number (kg/mm^2), standard deviation and coefficient of variance for all materials tested in low stress area.

Material	Treatment	N	Mean	SD	CV
Cerasmart	Static	10	68.48	2.22	3.24
	100k	10	69.32	2.23	3.21
	200k	10	68.12	2.44	3.58
e.max CAD	Static	8	517.01	8.46	1.64
	100k	10	504.58	16.17	3.21
	200k	10	479.48	10.33	2.16
Empress CAD	Static	10	449.82	31.52	7.01
	100k	10	483.27	19.89	4.12
	200k	10	461.51	28.13	6.1
Enamic	Static	8	198.93	11.94	6
	100k	9	216.63	22.49	10.38
	200k	8	231.96	29.51	12.72
LavaUlt	Static	10	105.99	2.02	1.9
	100k	10	108.97	4.83	4.43
	200k	10	107.82	6.5	6.03

The Vickers hardness number (VHN) of the material tested in static mode in low stress area ranged from 517.01 ± 8.46 kg/mm^2 for e.max CAD, to 68.48 ± 2.22 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 449.82 ± 31.52 kg/mm^2, followed by Enamic 198.93 ± 11.94 kg/mm^2, and Lava Ult 105.99 ± 2.02 kg/mm^2 respectively.

The Vickers hardness number (VHN) of the material tested after 100k cyclic loads in low stress area ranged from 504.58 ± 16.17 kg/mm^2 for e.max CAD, to 69.32 ± 2.23 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 483.27 ± 19.89 kg/mm^2, followed by Enamic 216.63 ± 22.49 kg/mm^2, and Lava Ult 108.97 ± 4.83 kg/mm^2 respectively.

The Vickers hardness number (VHN) of the material tested after 200k cyclic loads in low stress area ranged from 479.48 ± 10.33 kg/mm^2 for e.max CAD, to 68.12 ± 2.44 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 461.51 ± 28.13 kg/mm^2, followed by Enamic 231.96 ± 29.51 kg/mm^2, and Lava Ult 107.82 ± 6.50 kg/mm^2 respectively.

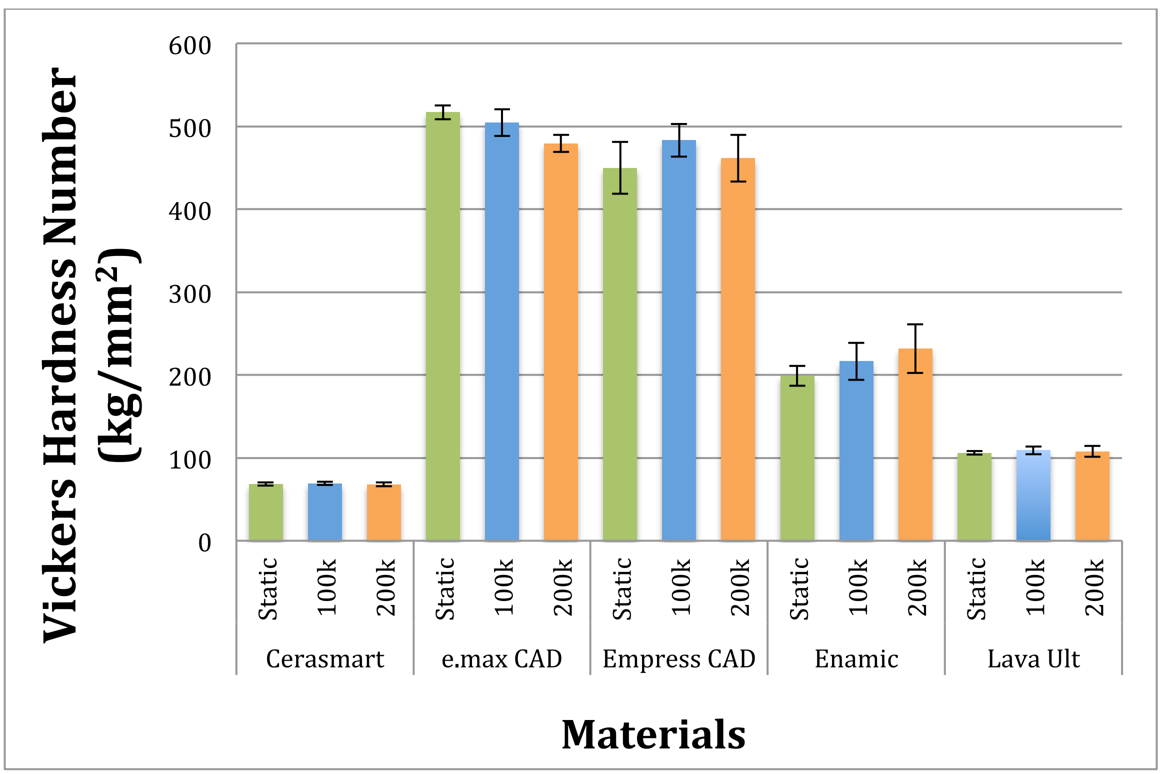

Figure 19: Mean Vickers hardness number (kg/mm^2) and standard deviation for all materials tested in low stress area.

3.2.1.7 VHN comparison in high stress area (B zone)

The mean Vickers hardness numbers of the five CAD/CAM dental restorative materials in high stress area are shown in Table 42.

Table 32: Mean Vickers hardness number (kg/mm^2), standard deviation and coefficient of variance for all materials tested in high stress area.

Material	Treatment	N	Mean	SD	CV
Cerasmart	Static	10	68.89	2.42	3.51
	100k	10	71.37	1.91	2.67
	200k	10	69.78	3.62	5.19
e.max CAD	Static	8	514.16	14.83	2.88
	100k	10	507.94	11.26	2.22
	200k	10	496.97	6.87	1.38
Empress CAD	Static	10	465.4	13.34	2.87
	100k	10	476.59	18.76	3.94
	200k	10	473.5	15.49	3.27
Enamic	Static	8	194.3	14.29	7.36
	100k	9	215.31	15.99	7.43
	200k	8	191.34	24.23	12.66
LavaUlt	Static	10	108.16	2.41	2.23
	100k	10	117.27	8.38	7.15
	200k	10	117.31	10.84	9.24

The Vickers hardness number (VHN) of the material tested in static mode in high stress area ranged from 514.16 ± 14.83 kg/mm^2 for e.max CAD, to 68.89 ± 2.42 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 465.40 ± 13.34 kg/mm^2, followed by Enamic 194.30 ± 14.29 kg/mm^2, and Lava Ult 108.16 ± 2.41 kg/mm^2 respectively.

The Vickers hardness number (VHN) of the material tested after 100k cyclic loads in low stress area ranged from 507.94 ± 11.26 kg/mm^2 for e.max CAD, to 71.37 ± 1.91 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 476.59 ± 18.76 kg/mm^2, followed by Enamic 215.31 ± 15.99 kg/mm^2, and Lava Ult 117.27 ± 8.38 kg/mm^2 respectively.

The Vickers hardness number (VHN) of the material tested after 200k cyclic loads in high stress area ranged from 496.97 ± 6.87 kg/mm^2 for e.max CAD, to 69.78 ± 3.62 kg/mm^2 for Cerasmart. Empress CAD had a Vickers hardness number (VHN) of 473.50 ± 15.49 kg/mm^2, followed by Enamic 191.34 ± 24.23 kg/mm^2, and Lava Ult 117.31 ± 10.84 kg/mm^2 respectively.

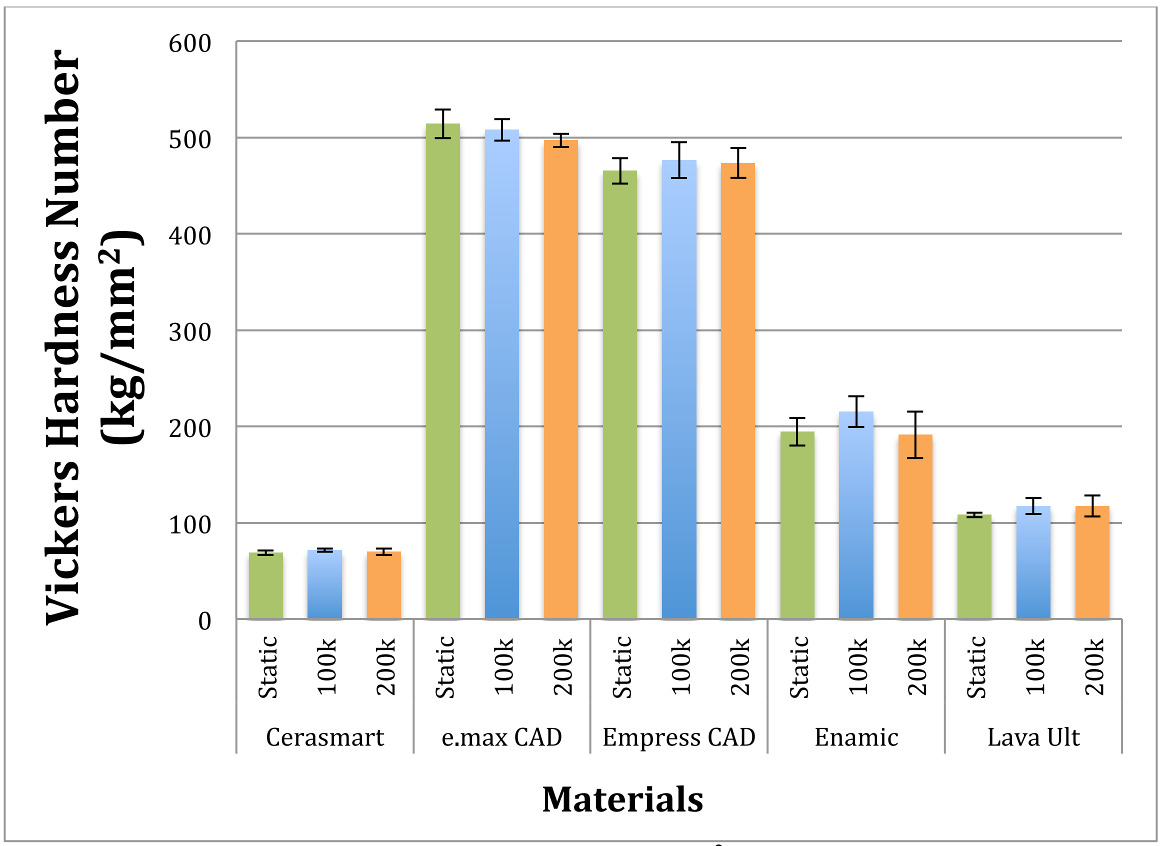

Figure 20: Mean Vickers hardness number (kg/mm^2) and standard deviation for all materials tested in high stress area.

3.2.2 Comparison of microhardness test of materials by treatment

The Vickers hardness number (VHN) of the five CAD/CAM dental restorative materials was determined, and then a comparison was done for treatments applied by using different statistical analysis that follows.

3.2.2.1 Lava™ Ultimate Restorative

Using different statistical analysis that is listed below compared Vickers hardness number of all treatments applied for Lava™ Ultimate Restorative.

Table 33: Tukey HSD test results of Vickers hardness number (kg/mm^2) by treatment for Lava™ Ultimate Restorative.

Abs(Dif)-HSD	Fatigued 100k	Fatigued 200k	Static
Fatigued 100k	-5.6897	-5.1347	0.3553
Fatigued 200k	-5.1347	-5.6897	-0.1997
Static	0.3553	-0.1997	-5.6897

Table 34: Connecting letters report of Vickers hardness number (kg/mm^2) by treatment for Lava™ Ultimate Restorative.

Level	Letter		Mean
Static		B	107.0750
Fatigued 100k	A		113.1200
Fatigued 200k	A	B	112.5650

3.2.2.2 IPS Empress® CAD

Using different statistical analysis that is listed below compared Vickers hardness number of all treatments applied for IPS Empress® CAD.

Table 35: Tukey HSD test results of Vickers hardness number (kg/mm^2) by treatment for IPS Empress® CAD.

Abs(Dif)-HSD	Fatigued 100k	Fatigued 200k	Static
Fatigued 100k	-17.077	-4.652	5.243
Fatigued 200k	-4.652	-17.077	-7.182
Static	5.243	-7.182	-17.077

Table 36: Connecting letters report of Vickers hardness number (kg/mm^2) by treatment for IPS Empress® CAD.

Level	Letter		Mean
Static		B	457.6100
Fatigued 100k	A		479.9300
Fatigued 200k	A	B	467.5050

3.2.2.3 Enamic®

Using different statistical analysis that is listed below compared Vickers hardness number of all treatments applied for Enamic®.

Table 37: Tukey HSD test results of Vickers hardness number (kg/mm^2) by traetment for Enamic®.

Abs(Dif)-HSD	100k	200k	Static
Fatigued 100k	-18.761	-15.016	0.021
Fatigued 200k	-15.016	-19.899	-4.862
Static	0.021	-4.862	-19.899

Table 38: Connecting letters report of Vickers hardness number (kg/mm^2) by treatment for Enamic®.

Level	Letter		Mean
Static		B	196.6125
Fatigued 100k	A		215.9722
Fatigued 200k	A	B	211.6500

3.2.2.4 IPS e.max® CAD

Using different statistical analysis that is listed below compared Vickers hardness number of all treatments applied for IPS e.max® CAD.

Table 39: Tukey HSD test results of Vickers hardness number (kg/mm^2) by treatment for IPS e.max® CAD.

Abs(Dif)-HSD	Static	Fatigued100k	Fatigued 200k
Fatigued 100k	-10.822	-0.939	17.096
Fatigued 200k	-0.939	-9.680	8.355
Static	17.096	8.355	-9.680

Table 40: Connecting letters report of Vickers hardness number (kg/mm^2) by treatment for IPS e.max® CAD.

Level	Letter		Mean
Static	A		515.5875
Fatigued 100k	A		506.2600
Fatigued 200k		B	488.2250

3.2.2.5 CERASMART™

Using different statistical analysis that is listed below compared Vickers hardness number of all treatments applied for CERASMART™.

Table 41: Tukey HSD test results of Vickers hardness number (kg/mm^2) by treatment for CERASMART™.

Abs(Dif)-HSD	100k	200k	Static
Fatigued 100k	-1.9691	-0.5741	-0.3091
Fatigued 200k	-0.5741	-1.9691	-1.7041
Static	-0.3091	-1.7041	-1.9691

Table 42: Connecting letters report for Vickers indentation hardness (kg/mm^2) by treatment of CERASMART™.

Level	Letter	Mean
Static	A	68.6850
Fatigued 100k	A	70.3450
Fatigued 200k	A	68.9500

3.3 Indentation crack measurement

Selected specimens were examined under the FESEM to measure the indentation crack length. Next two figures show the typical images of the indents found on the material surfaces.

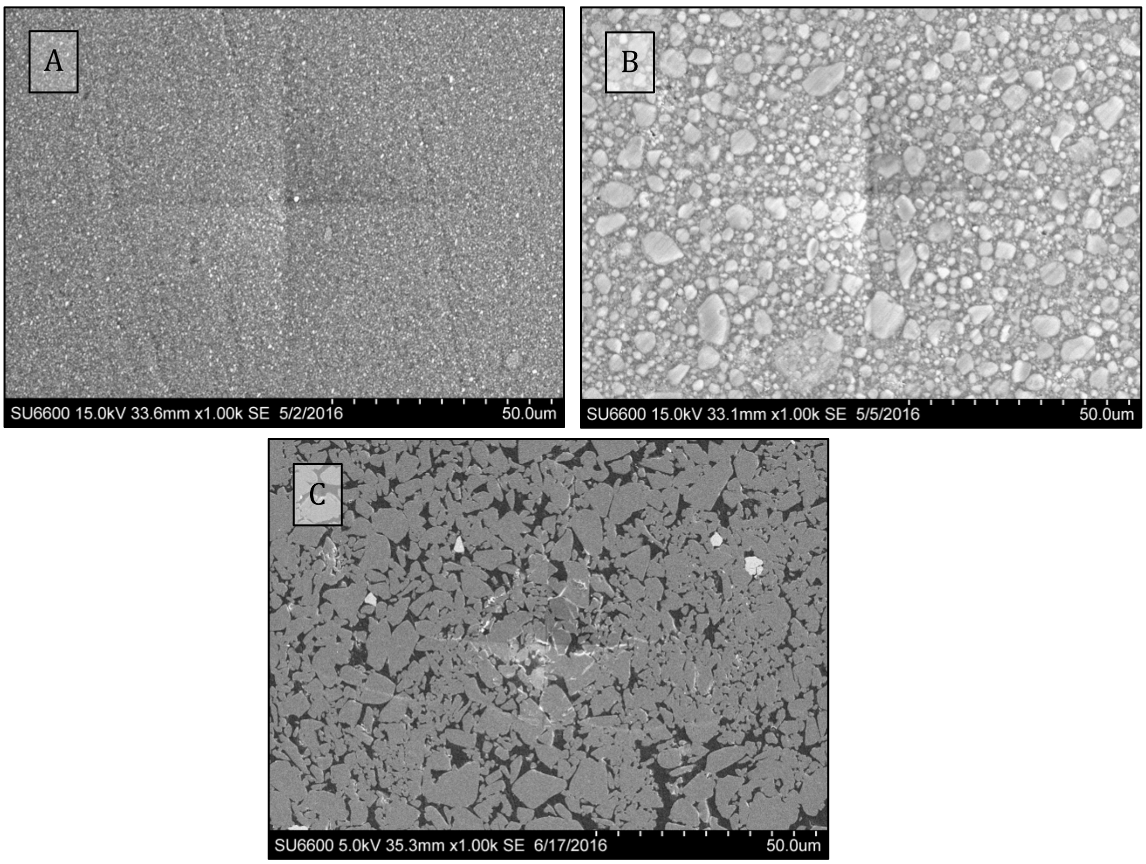

Figure 21: SEM images showing Vickers indentation site on different CAD/CAM materials surfaces with impression of weak indent shape and there is no clear appearance of crack. Images are from (A) CERASMART™. (B) Lava™ Ultimate Restorative. and (C) Enamic® block material.

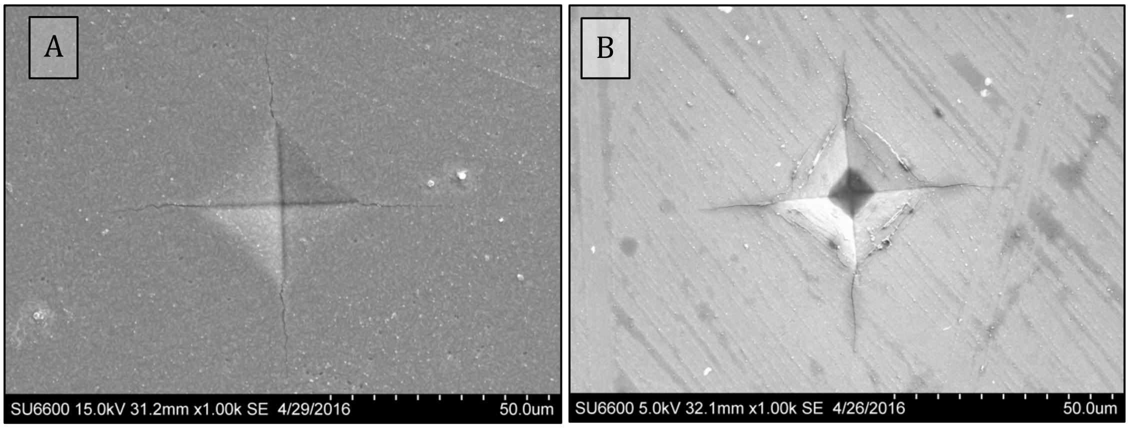

Figure 22: SEM images showing Vickers indentation site on the surface of (A) IPS e.max® CAD. (B) IPS Empress® CAD. With impression of clear indent and clear crack profile.

Figure 21 shows the SEM images of CERASMART™, Lava™ Ultimate Restorative and Enamic® block materials. No obvious crack can be found at the corners of most indents. In some indents of Enamic®, sparsely short cracks were found. Those materials were excluded from the calculation of fracture toughness (K_{1C}) by using the indentation fracture (IF) method because crack are not readily apparent for the experiment method.

Figure 22 was showing SEM images of CAD/CAM materials that were included in the experiment used to calculate fracture toughness (K_{1C}) with indentation fracture (IF) method because both indent and crack are clear. The crack length was measured from the center of the indent to the tip and equation from section 2.2.4 was used to calculate the indentation fracture toughness (K_{1C}), the results follows.

3.4 Fracture toughness (K_{1C}) using indentation fracture (IF) method

The fracture toughness of the two CAD/CAM dental restorative materials was determined by using indentation fracture (IF) method. The mean fracture toughness ($MPa \cdot m^{1/2}$), standard deviation, and coefficient of variation for each material were calculated.

Table 43: Mean indentation fracture toughness ($MPa \cdot m^{1/2}$), standard deviation and coefficient of variance for the materials tested by indentation fracture method.

Material	Treatment	N	Mean	SD	CV
e.max CAD	Static	16	1.41	0.09	6.52
	100k	18	1.38	0.16	11.67
	200k	19	1.29	0.06	4.38
Empress CAD	Static	13	1.38	0.14	10.27
	100k	17	1.24	0.09	7.59
	200k	18	1.26	0.08	6.59

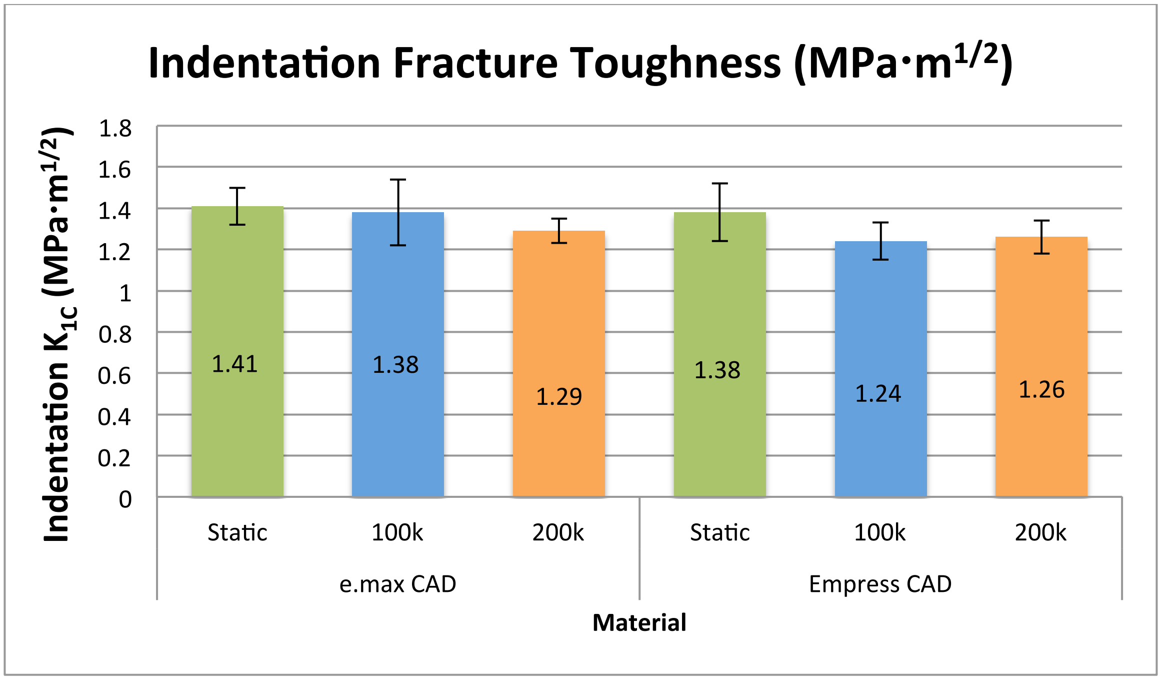

Figure 23: Mean indentation fracture toughness (MPa·m$^{1/2}$) and standard deviation for the materials tested by indentation fracture method.

The indentation fracture toughness of the material tested in static mode ranged from 1.41 ± 0.09 MPa·m$^{1/2}$ for e.max CAD, to 1.38 ± 0.14 MPa·m$^{1/2}$ for Empress CAD.

The indentation fracture toughness of the material tested after 100k cyclic loads ranged from 1.38 ± 0.16 MPa·m$^{1/2}$ for e.max CAD, to 1.24 ± 0.09 MPa·m$^{1/2}$ for Empress CAD.

The indentation fracture toughness of the material tested after 200k cyclic loads ranged from 1.29 ± 0.06 MPa·m$^{1/2}$ for e.max CAD, to 1.26 ± 0.08 MPa·m$^{1/2}$ for Empress CAD.

3.4.1 Comparison between fracture toughness by single edge pre crack beam (SEPB) method and indentation fracture (IF) method

After both IPS e.max® CAD and IPS Empress® CAD were tested for fracture toughness values, by using two methods single edge pre-crack beam (SEPB) and indentation fracture (IF), results were compared and found that fracture toughness had lower values when IF method was used compared to SEPB method.

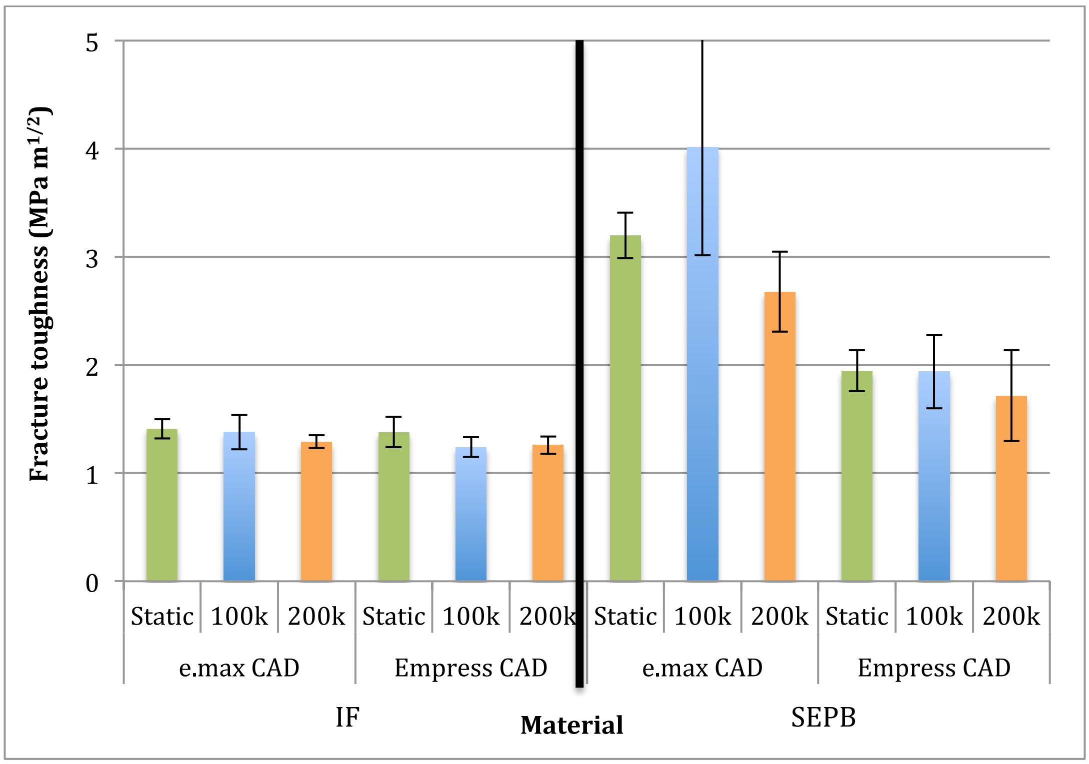

Figure 24: comparison of mean fracture toughness (MPa·m$^{1/2}$) by SEPB on the right and by IF on the left.

SEPB= Single edge pre crack beam
IF= Indentation fracture

3.5 Microstructural analysis by SEM

Selected IPS e.max® CAD specimens were examined under FESEM for microstructural analysis on the fractured surface. Figure 24 shows the low magnification of the fracture surface of static and after 200k cyclic loading fatigues. On the surface of 200k cyclic loading fatigue (Fig. 25-B), a significant secondary electron contrast can be easily identified between the high stress and low stress areas, while on the fracture surface of static specimens, such contrast was not found obviously (Fig. 25-A).

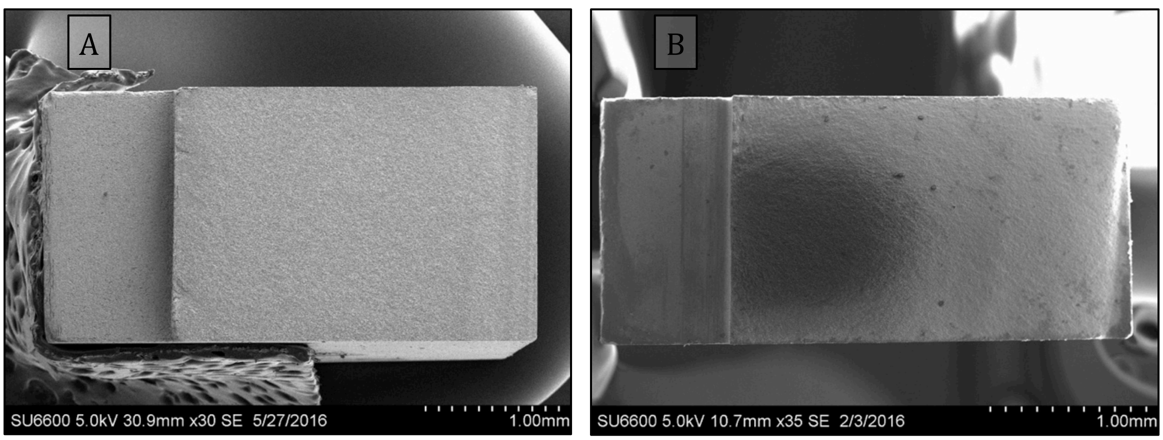

Figure 25: SEM images for etched IPS e.max® CAD specimens showing the stress areas on the fracture surface after three point bending test. In the group of (A) static. (B) after 200k cyclic loading.

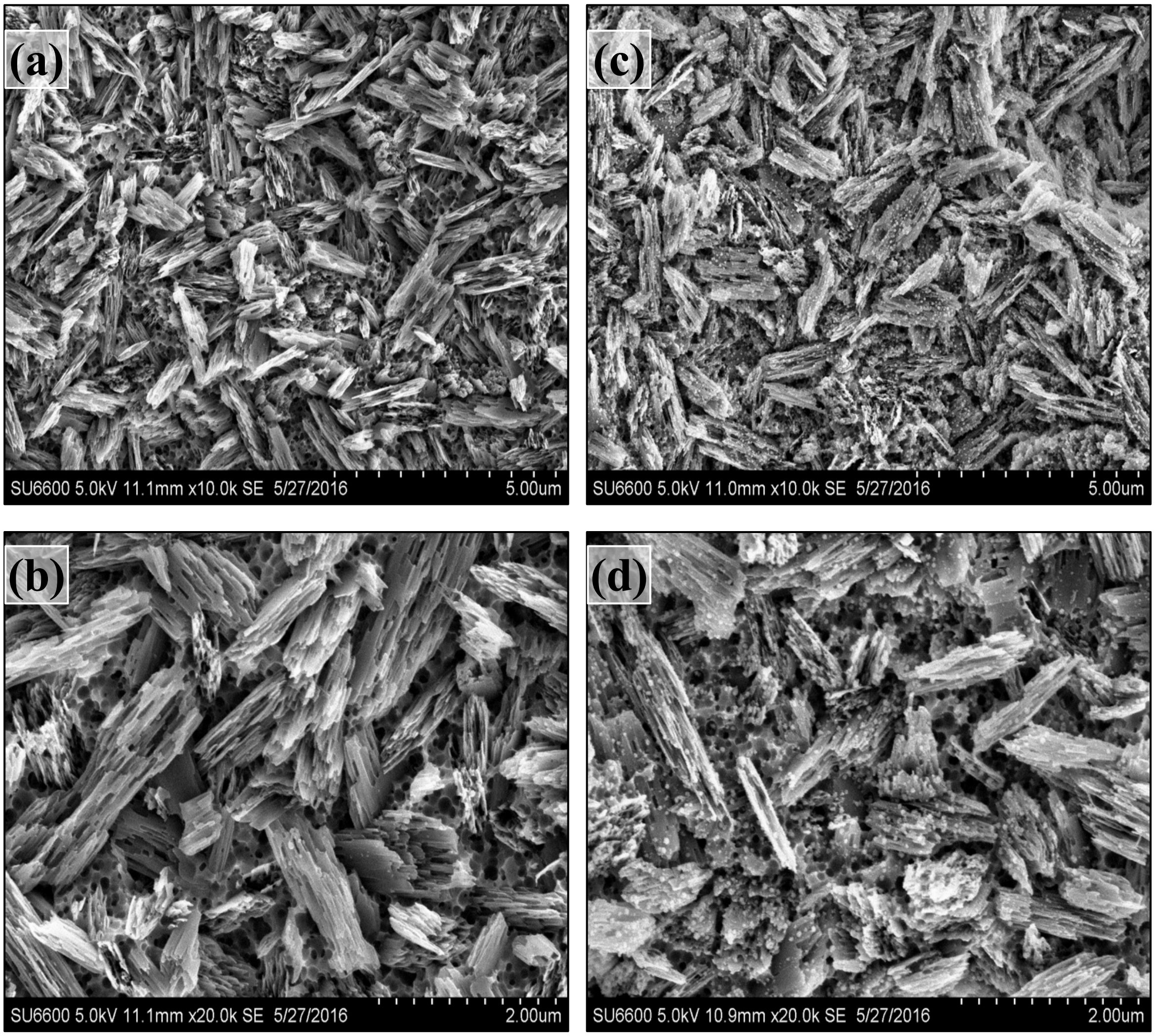

Figure 26: SEM images for etched IPS e.max® CAD specimens showing a high magnification ×10k of stress zone area on the top images. Higher magnification ×20k was showing on the bottom images for the same area. Images are of (a and b) static group specimen. (c and d) specimen from after 200k cyclic loading group.

After etching two specimens of IPS e.max® CAD with 9% Hydrofluoric acid for 2 minutes they were cleaned ultrasonically with distilled water for 3 minutes. One specimen was from static group, the other was from group after 200k cyclic loading, general new crystalline phase changes was observed due to fatigue, that can contribute why fracture toughness (K_{1C}) is different after fatigue treatment. A spherical shape particle was observed on the 200k fatigued specimen had a reinforce effect that most probably increased the material resistant property on the specimen at the stress area around the notch. (Fig. 26-a, 26-b)

Chapter 4. DISCUSSION

The results of the tests performed help answer several questions about the mechanical performance of chairside CAD/CAM dental restorative materials, while at the same time, opening up avenues for more in-depth investigations to be conducted.

Flexural strength is one of the most important mechanical properties for dental material, has been widely studied and testing it in vitro considered to be the standard, its clinical relevance has been evaluated. However, its value is not inherent because it depends on the material condition and the way the test was done. It is believed that when a material restoration applied to an adhesive interface, it should consider both strength and defects at or near the interface and should therefore be more important for characterization of interfacial fracture resistance than conventional bond strength.(32,51)

Fracture toughness (K_{1C}) is an important characterization value for materials. Its value reflects the ability of the material to resist the crack initiation and unstable propagation.

When a material has a high fracture toughness (K_{1C}) value that reflects the better material behavior of resisting crack propagation. Unfortunately, dental ceramic materials mostly contain crystalline particles embedded in a glassy matrix that represent its fracture toughness (K_{1C}) with a low value even lower than polycrystalline ceramics because these glassy matrices are very brittle. Nevertheless the ceramic materials are widely used in dental practice because of their excellent esthetic restorative outcomes.

There is a verity wide range of methods to measure the fracture toughness of ceramic materials. According to ASTM C1421 they standardized three methods for K_{1C} measurement. One method is SNPB that was used in this study due to its applicability with small size of material specimens. In addition, we used the indentation fracture method for two materials in the study, IPS e.max® CAD and IPS Empress® CAD, because those materials introduces clear crack track after Vickers indentation was applied, the other three remaining materials tested in the study lack the ability to appear extend crack flaw due to the material particle contents. In general the indentation method can be used on hard and brittle materials and hard to achieve in some of them due to the disability of seeing a clear crack track. It was reported that the indentation method are extremely material dependent.(40) For example, CAD/CAM Enamic® material after Vickers indentation was done and by examining the material under the FESEM, the SEM image showed surface of the material with impression of weak indent shape and there was no clear appearance of crack (Fig. 20-C). A high magnification SEM image for the same surface location for the material shows random appearance of small cracks that extended from one particle to another one, which is extremely hard to track and its not compatible with the equation used in this study.

In addition, the crack length of some brittle materials may extend after indentation test. This post-indentation cracking will cause a lower value of the K_{1C} than actual (42).

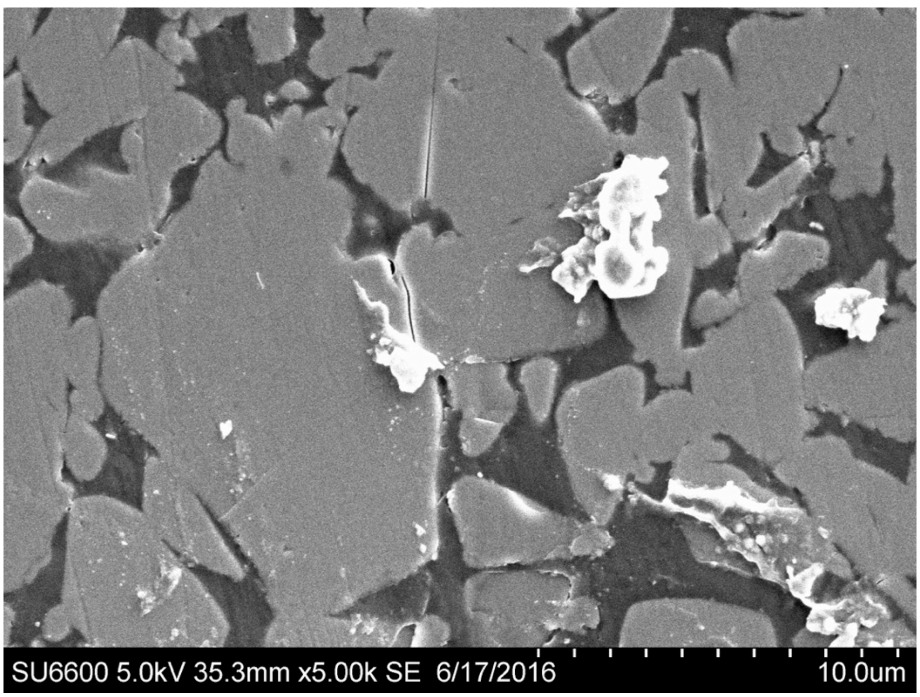

Figure 27: SEM image showing small crack extensions at the indent corner of Vickers indentation site on Enamic® material surfaces.

In the present study, the result of fracture toughness (K_{1C}) in static mode showed that IPS e.max CAD 3.2 ± 0.21 MPa·m$^{1/2}$ had higher values from all other tested materials.

The results for fracture toughness (K_{1C}) by Single-edge-pre-crack beam of static group for IPS e.max® CAD 3.2 ± 0.21 MPa·m$^{1/2}$, IPS Empress® CAD 1.95 ± 0.19 MPa·m$^{1/2}$, and Enamic® 1.92 ± 0.29 MPa·m$^{1/2}$ are higher than the data from manufactures' in-house testing reports. We speculate that is due to the different method used to test the K_{1C} value because they may have tested larger dimensioned specimen, but in this study the block size is one of the limitations and also they may have used a different testing conditions. In addition, IPS Empress® CAD K_{1C} value result for static group agree with one of the studies.(23) On the other hand, Lava™ Ultimate Restorative 2.00 ± 0.60 MPa·m$^{1/2}$ agree with the manufactures' published data, while CERASMART™ 1.65 ± 0.61 MPa·m$^{1/2}$ which was no published data exist on the material as far as we know.

In this study, the results for K_{1C} after cyclic loading were compared. They showed that IPS e.max CAD 4.02 ± 1.00 MPa·m$^{1/2}$ had higher values than all other tested materials after 100k cyclic loading, while CERASMART™ 3.14 ± 0.39 MPa·m$^{1/2}$ had higher values among other materials after 200k cyclic loading. There were no published studies as far as we know comparing fracture toughness (K_{IC}) after cyclic loading for specimens prepared from various materials with a pre-crack. However, some studies did a comparison other than the materials used in this study such as Lava All-Ceramic System (Lava, 3M ESPE, Seefeld, Germany) YTZP core ceramics by using indentation fracture method (52).

Contrary to our findings, in the literature review, we found that fracture toughness (K_{1C}) measured by using the indentation fracture method gave higher values and that could be due to several factors that includes the timing the crack measurement was done, and the way used to measure the crack length. In most of the previous studies the optical microscope of the hardness tester machine was used instead of SEM for measuring the crack length of the indentation. (51) An inaccurate measurement will overestimate the value for fracture toughness (K_{1C}), because the minor fracture crack track could not be easily found under a low magnification optical microscope. The delayed measurement, causing crack propagation of the crack length, was also an important factor that lowered the calculated value of fracture toughness (K_{1C}). Therefore, our current study we got a lower value of fracture toughness (K_{1C}) by using the indentation fracture (IF) method.

Chapter 5. CONCLUSIONS

Within the limitations of this in-vitro study, the following conclusion can be drawn:

1. The mean fracture toughness of e.max CAD material is significantly higher than other CAD/CAM restorative materials tested. ($p<0.0001$)
2. In general, CAD/CAM materials tested after 100k cyclic loading, showed significantly higher fracture toughness ($p=0.0006$) than in static mode.
3. In general, CAD/CAM materials tested after 200k cyclic loading, also showed significantly higher fracture toughness ($p=0.0459$) than in static mode.
4. After 100k cyclic loading, e.max CAD material, showed significant higher fracture toughness than in static mode. ($p=0.0125$)
5. Cerasmart and Lava Ultimate CAD/CAM materials showed significant higher fracture toughness after 100k and 200k cyclic loading than in static mode.
6. Empress CAD material showed higher fracture toughness after 100k and 200k cyclic loading, but not significant. ($p=0.998, 0.273$)
7. In general, after 100k cyclic loading all CAD/CAM materials tested showed significantly higher Vickers hardness number (VHN) than the static group.
8. In general, after 100k cyclic loading for all CAD/CAM materials tested showed high stress area significantly higher Vickers hardness number (VHN) than the low stress area before cyclic loading. ($p=0.0192$)
9. In general, there is no significant difference between the two stress areas for tested materials. ($p=0.53$)

10. In general, after 100k and 200k cyclic loading, fracture toughness values tested by indentation method were significantly lower. (p=0.0001, 0.0067)
11. Fracture toughness tested by indentation method in our study showed no significant difference between low stress and high stress zones. (p=0.319)

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Curriculum Vitae

Alaa Kensara was born in Jeddah, Saudi Arabia in 1982. He attended King Abdul Aziz University- School of Dentistry in Jeddah, Saudi Arabia, where he earned his Bachelor of Dental Surgery degree in July of 2006, then he completed one year in training as an internship program. Immediately following, in September of 2007 he was hired as a faculty in King Abul Aziz University- School of Dentistry. In July of 2010, he joined the Prosthodontics Division at Boston University Henry M. Goldman School of Dental Medicine, where he pursued a five-year program culminating in a Certificate of Advanced Graduate Studies in Prosthodontics and a Doctorate of Science in Dentistry upon successful completion of the dissertation herein. He can be reached at the following e-mail address: akensara@bu.edu.