THE MECHANICAL PROPERTIES OF

FULL-CONTOUR ZIRCONIA

by

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DEDICATION

This thesis is dedicated to all the people who stand beside me in my life:

To my wonderful wife, Sura, and my lovely daughter, Leen, for their love and patience during my studies.

To my great parents, Dr. Uday and Dr. Siham, for their guiding support and motivation. To my brothers and sister, Dr. Firas, Dr. Ahmed, and Sura.

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INTRODUCTION

High esthetics, excellent biocompatibility and high flexural strength have fueled public demand for all-ceramic instead of porcelain-fused to metal crowns. However, dentists face challenges when it comes to the decision as to the most appropriate material for each patient since all-ceramic crowns are associated with some disadvantages. Ceramic is brittle and has low tensile strength and fracture toughness due to unavoidable inherent imperfection as they potentiate cracks when subjected to stress. The most common complication with all-ceramic crowns is fracture.[1] This has resulted in a search for ways to increase the fracture strength such as incorporating new materials, reducing particles size, utilizing different veneering techniques or modifying the processing technique.[2] Slip-cast, heat pressed, and machined are common processing techniques for ceramic crown fabrication. The slip-cast technique shows large spectra of strength distribution resulting in a low-Weibull modulus while processing through the CAM/CAM technology increases the Weibull modulus of the material.[3] Veneering a CAD/CAM designed core provides high strength with high optical quality, which is commonly being used in the recent dentistry. However, veneering material is usually weaker than the core material which leads to the typical failure pattern, chipping of the veneer layer.[4]

Some manufacturers have introduced a new approach by designing a full contour crown (no veneering) from a CAD/CAM ingot to avoid the problem from veneering. IPS e.max CAD (lithium disilicate glass-ceramic) and IPS Empress CAD (leucite) are examples of these crowns. The flexural strength of lithium disilicate glass-ceramic and leucite glass ceramic are around 350 MPa and 160 MPa respectively.[5] These materials are suitable for anterior and premolars restorations only because the strength is not sufficient to withstand occlusal forces generated by posterior teeth.

Although all-ceramic (leucite and lithium disilicate) crowns have a good success rate, researchers continued to search for a tougher material that can replace the metal framework. This led to development of dental zirconia which is currently one of the toughest ceramic materials. It was described as ceramic steel by Garvie[6] as it has a flexural strength of 1000 MPa.[5] Zirconia was first used in dentistry in 1990s although first application in Orthopedics occurred much earlier in 1969.[7] Being white-colored and high in strength, it is commonly used as framework when esthetic is highly demanded and heavy occlusion is expected.[8] Zirconia framework is usually veneered with porcelain, leucite reinforced glass ceramic, or lithium disilicate reinforced glass ceramic. Because of the possibility of chipping the veneer layer, the idea of fabricating a crown made entirely from zirconia was proposed. That would merge the strength and the esthetics of the zirconia material. However, the potential for wear of opposing dentition,[9] the potential for loss of strength during aging,[8] and difficulty in shade matching with natural teeth due to its opacity become problematic when considering zirconia for fabrication of full contour crowns.

Currently, research has been conducted to enhance the optical quality and strengthening the structure of zirconia to allow fabrication of full zirconia crowns. One of the common strengthening methods is transformation toughening, which is done usually by adding 3 mol% yttria to the tetragonal zirconia polycrystalline. The tetragonal-monoclinic phase transformation, accompanied with 3 percent to 4 percent volume expansion, will arrest crack propagation and increase the toughness.[10] It was reported that aging has little or no influence on the mechanical properties of the contemporary wellmanufactured yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) materials,[8] and these materials are more stable than the one used in the early 1980s in orthopedic studies which showed failure with aging.[3,12]

Several companies have been working on different processing techniques to improve the esthetic result of zirconia. That has resulted in a new generation of zirconia with a higher translucency than the traditional zirconia used for core fabrication only. In this paper, we referred to zirconia with higher translucency as “translucent zirconia,” and to the zirconia for core fabrication as “non-translucent traditional zirconia.”

However, the mechanical properties of translucent zirconia still need verification. Also, due to the high strength of these translucent Y-TZP zirconia materials, it is proposed that less tooth reduction is needed to achieve the same or greater overall strength in the crown when compared with the reduction needed for lithium disilicate crowns. Still, the scientific data supporting the claim is still lacking. Therefore the objectives of this study are:

1. To compare the flexural strength, flexural modulus, and fracture toughness of specimens fabricated from recently marketed translucent full contour zirconia, traditional zirconia, and lithium disilicate glass ceramic.

2. To compare the load-to-failure of crowns fabricated from recently marketed translucent full contour zirconia, traditional zirconia, and lithium disilicate glass ceramic at their recommended tooth reduction thickness.

HYPOTHESES

Null Hypothesis

1) Translucent zirconia will have lower flexural strength, fracture toughness, and fracture toughness compared with traditional non-translucent zirconia.

2) The crowns fabricated from translucent zirconia at the recommended reduction thickness have lower load-to-failure value than the non-translucent traditional zirconia and lithium disilicate.

Alternate Hypothesis

1) Translucent zirconia will have the same or higher flexural strength, fracture toughness, and fracture toughness compared with traditional non-translucent zirconia.

2) The crowns fabricated from translucent zirconia at the recommended reduction thickness will have the same or higher load-to-failure value than the non-translucent traditional zirconia and lithium disilicate.

This study provides the fundamental understanding of the fracture behavior of the translucent zirconia.

REVIEW OF LITERATURE

CERAMICS

The term ceramic is derived from the word “keramos” that means “pottery” in Greek. Ceramic is not a new material in dentistry. It was first introduced in dentistry in 1789, and the first ceramic crown was placed by Charles Land, a French dentist.[13] In general, this material is strong in compression, but brittle and weak in tension. In contrast, metal is a ductile material. The type of bond between the atoms is responsible for brittleness and ductility.[14] Ceramic consists of glass matrix and crystals. Glass is responsible for the optical quality, and crystals are responsible for the strength. The greater the glass content, the higher the esthetics; the greater the amount of crystals, the greater the strength and opacity of the ceramic. However, the glass phase is the weak part, in which cracks propagate[15] leading to restoration failure. The properties of the ceramic count on the amount of crystals and the glass content, the interaction between them, the crystal size, and the processing technique.[14,16] Etchability of ceramic is an advantage; it offers micro-retention for the adhesive to penetrate.

Dental ceramics can be categorized by structure[14] into glass-based systems; glass-based systems with fillers; interpenetrating phase ceramics, and polycrystalline solids.

GLASS-BASED SYSTEMS

These consist mainly of aluminosilicate glass, which is highly esthetic and mimics natural teeth color. It has low flexural strength, ranges from 60 MPa to 70 MPa that can be used as veneering material for metal or ceramic. This material is called glassy porcelain.

GLASS-BASED SYSTEMS WITH FILLERS

These have the same materials as the previous category but have different amounts of crystals. Leucite and lithium disilicate are the primary crystals used today. This category includes:

- Low leucite content feldspathic glass ceramic.

This material is called feldspathic porcelain. It has a composition similar to Category 1 glass but contains a greater amount of crystals, which makes it stronger. However, due to large particles of 100-μm size, the material still shows the low fracture resistance and abrasion potential to enamel. Given the high content of fluoroapatite crystals,[14] feldspathic porcelain is the typical material used in a veneering core system because of its highly esthetic match for the shade of a natural tooth.

- High leucite content reinforced glass ceramic.

This kind of ceramic is made by increasing the potassium content (to 50 percent) to increase the mechanical strength while maintaining the optical quality. The common leucite-reinforced ceramic brands are IPS Empress (Ivoclar Vivadent), and OPC (Pentron). IPS Empress was developed at University of Zurich, Zurich, and was introduced to the dental market in 1990.[17] It has a 160-MPa flexural strength,[5] with crystals size of 1.5 µm to 2.6 µm that grow evenly in a multistage process.[18] OPC ceramic material has crystals of 1.9 µm to 6.6 µm in size,[19] and it has a biaxial flexural strength of 153.60 MPa, slightly more than IPS Empress (134.4 MPa).[20] However, Cattell et al. in 1999 showed no significant difference between Empress and OPC material that ranges from 135.8 MPa to 139.1 MPa.[19]

Machinable blocks of leucite-reinforced glass ceramic are available, such as Empress CAD (Ivoclar). The machinable and pressable types are shown to have higher fracture resistance, and are reported to have good clinical results when used for veneers, inlays, onlays and anterior crowns.[16-21]

Frankenberger et al.[22] conducted a controlled prospective clinical trial of IPS-Empress inlays and onlays. Ninety-six restorations were placed in 34 patients and assessed according to modified US Public Health Service (USPHS) criteria. After 6 years, the survival rate was 93 percent, although 69 percent of the restorations were available for evaluation. Good marginal quality was noticed in 43 percent of the restorations.

Similarly, Lehner and others[23] conducted a clinical trial involving 138 inlays and 17 onlays in which 60 percent of them were placed on molars. The restorations were also evaluated according to modified United States Public Health Service (USPHS) criteria, and the surviving rate was 95 percent after 6 years. The percentage went down to 91 percent after one more year.

- Lithium disilicate glass ceramics

With the purpose to increase the strength of dental ceramic while maintaining the optical quality, lithium disilicate glass ceramics were introduced by Ivoclar as IPS Empress II. It contains glass matrix and 70-percent micron-size lithium disilicate crystals. The crystals are made by adding lithium oxide to the aluminosilicate glass, which acts as a flux to decrease the melting temperature of the material.[14] Refining crystals size and increasing the amount of crystals lead to flexural strength of 360 MPa, which is about three times stronger than leucite glass ceramic.[24] Lithium disilicate crystals have low refractive index that provides translucency even with a large crystal content. IPS e.max (Ivoclar) was introduced in 2005 with more enhanced properties than IPS Empress II, including better physical properties and esthetics.[25] This type of ceramic can be used for fabrication of a three-unit unit bridge in the anterior region that can extend up to the second premolar.[26-27] CAD/CAM blocks for e.max are available under the name of IPS e.max CAD. They come partially sintered, which calls for further heat treatment to complete the sintering and full growth of the crystals. Because of the low thermal expansion, no consideration is made for the size of the crown before milling, given the minimum shrinkage in the material during processing. In general, CAD/CAM blocks were reported to have better mechanical properties than the pressable system because of the standardized manufacturing process.[28] These blocks can be used for posterior crowns and three-unit FPDs.[29-30] Due to the enhanced mechanical properties and good esthetic results, lithium disilicate ceramic crowns are widely used now with the success well-documented in the literature. Taskonak and Sertgöz evaluated the clinical performance of IPS Empress 2 for single crowns. Twenty anterior or posterior crowns were placed and evaluated after 2 years according to US Public Health Service criteria. The crowns showed no recurrent caries or fracture during the two years.[30] Interestingly, Wolfart et al.[31] conducted an *in-vivo* study to evaluate 33 IPS e.max press anterior and posterior FPDs. After 8 years, the survival rate Kaplan-Meier statistic was 93 percent. There were two fractured and two debonded restorations, and the latter only needed re-cementation.

INTERPENETRATING PHASE CERAMICS

This type of ceramic involves the In-Ceram family (Vivadent). They are made by fabricating porous matrix, and then filled with lanthanum aluminosilicate glass. In-Ceram Spinell (alumina and magnesia matrix) is the most translucent with flexural strength of 350 MPa, which can be used for anterior crowns. In-Ceram Alumina has 450-MPa flexural strength but lower translucency than the previous one. In-Ceram Zirconium (alumina and zirconium matrix) has 650-MPa flexural strength and poor translucency.[14] The last two are usually veneered by porcelain due to their opacity.[32-33] An *in-vitro* study was conducted by Al-Wahadni et al. to compare the fracture strength of In-Ceram Alumina and IPS Empress 2 crowns using glass ionomer and resin cement. The In-Ceram Alumina showed higher resistance to fracture than Empress 2, with no statistical difference between the two cements.[34] The same materials were evaluated by Quran and others in an *in-vitro* study; In-Ceram Alumina crowns had mean fracture load of 941.8 N compared with 534 N for IPS Empress 2.[35]

POLYCRYSTALLINE SOLIDS

These ceramics are made by directly sintering crystals together without the glass phase to form a dense, air-free polycrystalline structure. Procera was the first dental application for fully dense polycrystalline material. It has a flexural strength of 600 MPa.[36] The other type of polycrystalline ceramic is zirconia. It has flexural strength of approximately 900 MPa to 1100 MPa, and fracture toughness of 8-10 MPa $m^{1/2}$.[37]

ZIRCONIA AS CORE MATERIAL FOR CROWNS AND FPDs

Zirconia is the toughest dental ceramic available in dentistry. The particle size is 0.1 µm to 0.5 µm.[14] White in color and possessing relatively great strength, it has been used to fabricate crowns and FPDs frameworks as an alternative to metal. Clinical studies have not shown a problem with zirconia frameworks.[38-39] The most common failure reported of zirconia restorations is due to the chipping of the veneering material.[40] Dental zirconia is not pure zirconia. It contains additives to increase the toughness through the “transformation toughening” mechanism. Adding additives stabilizes the tetragonal phase of zirconia in room temperature. Stress concentration on the tip of the crack leads to transformation of tetragonal to monoclinic, which is associated with 4-percent expansion, and the stress due to the expansion hinders the propagation of cracks.[10] Yttria (Y_2O_3), magnesia (MgO), and ceria (CeO_2) are common oxides added to zirconia for a toughening mechanism.[41] Yttria is usually used over the other oxides because the transformation toughening mechanism is fully active yttria-stabilized zirconia, while it is less pronounced in ceria/magnesia-stabilized zirconia.[42]

Zirconia comes in the form of porous or dense CAD/CAM blocks. The porous block is widely used because it is not fully sintered, which allows it to be milled more easily. The restorations should be milled oversized by about 25 percent to compensate for the shrinkage associated with the final sintering. Restorations can be milled from a dense fully sintered block directly to the desired size; howver, it would take 2 hours to mill one unit, while it takes 30 minutes to 45 minutes to mill three-unit FPDs from porous blocks.[14]

Kim et al.[43] compared the fracture load of zirconia-based anterior crowns with different core thicknesses. All the crowns were veneered by IPS e.max Ceram (Ivoclar Vivadent). Crowns with 0.5-mm core thickness showed a mean fracture load of 2126.9 N±576.9, and 3179.3 N±1137.7 for those with 0.7-mm core thickness. A clinical performance of zirconia substructure was investigated by Peláez [44] Twenty zirconia-based FPDs were placed in 17 patients and evaluated after 3 years for surface and color, anatomic form, and margin integrity. Lava systems (3M ESPE) were used to fabricate the frameworks and veneered by Lava Ceram (3M ESPE). By the end of the study, the survival rate was 95 percent; two cases suffered chipped veneers and one biological complication. Interestingly, no framework fracture was noted.

Similarly, Raigrodski et al.[39] conducted a three-year clinical study to assess the fracture resistance and marginal integrity of 17 zirconia-based FPDs, veneered by porcelain. No failure in the frameworks was observed, and 5 restorations had their veneers chipped. The two later studies infer zirconia is appropriate material to be used for framework fabrication, and chipping veneers is still a common problem.

FULL-CONTOUR ZIRCONIA

The most common problem associated with veneered zirconia crowns is still veneer chipping.[4,40] As a result, an idea has been generated to fabricate a full-contour crown made entirely from one type of ceramic material. Full-contour crowns are already available in lithium disilicate, IPS e.max CAD, and for leucite ceramic, IPS Empress CAD. Machined restorations are believed to have better physical properties than the hot-pressed system due to the standardized manufacturer's process.[28] Another idea has been proposed to fabricate a full-contour crown made entirely from zirconia. Before zirconia can be used to fabricate a full anatomical crown, white opaque zirconia must be processed in a way to increase its translucency. Recently, several companies have advertised full-contour zirconia as a tough esthetic restoration. However, none has mentioned the processing technique that enhances the translucency or the impact on the mechanical properties. The idea of translucent full zirconia crowns is new, and not much is published about this type of crown. Johansson et al.[45]conducted *in-vitro* study to evaluate the fracture strength of monolithic translucent zirconia. Two brands of monolithic translucent zirconia were compared with veneered same-brands translucent zirconia, veneered non-translucent traditional zirconia, and monolithic lithium disilicate. Crowns were made, thermocycled 5000 cycles, and cemented on polyxymethylene resin dies with resin cement. The crowns were cyclically pre-loaded at a 10-degree angle for 10,000 cycles in a wet environment and then loaded to fracture. The two monolithic translucent zirconium brands showed a significantly higher fracture strength (2795 N to 3038 N) compared with other groups (1480 N to 2229 N).

Marchack et al.[46] presented a clinical report of complete- and partial-contour zirconia designs. He placed two full anatomical zirconia crowns, one buccally veneered zirconia crown, and one buccally veneered FPD. Over two years, no complication was observed.

ENHANCING THE OPTICAL QUALITY OF FULL-CONTOUR ZIRCONIA AND ITS IMPACT ON STRENGTH

In the literature, there are few processing techniques mentioned by researchers which led to increased translucency in the processed zirconia. Adding titanium oxide to yttrium stabilized zirconia was investigated by Radford and Bratton, and it was reported to be effective in densifying yttria-stabilized zirconia.[47] Tsukuma studied the effect of TiO2 on the transparency of zirconia, instead of translucency. He added 10 mol% TiO2 to 8 mol% yttria-zirconia powder and sintered it to 1430 °C for 12 hrs and 1630 °C for 7 hrs.[48] The x-ray diffraction showed that TiO2 dissolved in ZrO and formed a solid solution, but the grain size in TiO_2-doped zirconia was larger than in TiO2 undoped. That indicates that TiO_2 stimulates grain growth during sintering. It was found that the addition of TiO_2 provides a fairly high transmittance to the zirconia. Moreover, the pressure associated with TiO_2-adding technique led to pore migration, which is thought to increase the transparency and the strength as well.

Hot isostatic pressing (HIP) is a processing technique used to increase the translucency.[48-49] In this technique, the zirconia powder is heated by a heating coil and pressed at the same time. The pressure eliminates pores in the sintered material, but results in increased grain size,[48] which in turn deteriorates the mechanical and optical properties due to a reduction in grain boundaries.[50]

Alternatively, spark plasma sintering (SPS) is used to avoid the problems associated with the HIP technique. In SPS, a high density current flux runs through the sample and the die to provide the required heat while pressure is applied. This technique allows the use of a sintering temperature (~1200°C) and reduced heating and cooling time, thus minimizing the amount of grain growth and maintaining the nanostructure of the material.[51] A high pressure version of this technique is able to produce dense materials of less than 20-nm grain size.[52] That will lead to an elimination or a decrease in the pores in the material while creating more grain boundaries, so a tougher material will be obtained.

Casolco et al., Alaniz et al., and Anselmi-Tumburini et al. were able to change the shade of zirconia.[51,53,54] A vacuum and graphite die were used in the SPS technique to provide a thermally reduced environment. This led to oxygen vacancies, which are called color centers. These vacancies absorb the light and result in a yellow-brown coloration. Annealing in oxidizing atmosphere diffuses back oxygen and reduces those color centers. Holding time at 1200˚ C during sintering is responsible for the level of coloration.

However, the impact of these techniques on the mechanical properties of full-contour zirconia is not well investigated, and only a few studies have tested this type of material.

METHODS AND MATERIALS

Four translucent zirconia brands were selected to be compared with a non-translucent traditional zirconia brand (1st control group), and lithium disilicate (2nd control group) (Table I).

BAR SAMPLES PREPARATION

Twelve bar samples of each material were made from the CAD/CAM material blocks and disks using a cutting machine (Isomet 1000, Buehler, IL) (Figure 1). Samples with final dimensions of 20 (±0.3) x 1.8 (±0.1) x 5 (±0.1) mm3 were made.[55] Six of them were tested for flexural strength and modulus, and the other six for fracture toughness. Due to the shrinkage associated with sintering zirconia, the zirconia samples were cut oversized by a percentage specified by the manufacturers (ranging from 24.5 percent to 25 percent) (Table II, Figure 2). For fracture toughness bars, a final notch dimension of 2 (±0.5) mm was used. First, a primary notch of 2.5 (±0.2) mm in depth was machined at the mid-span of the zirconia bars before sintering using a 0.2-mm thick diamond cutting band (Exakt 300, EXAKT Technologies, Oklahoma City, Oklahoma), then a secondary V shape notch was cut at the tip of the primary notch using a sharp steel blade. After sintering, the notches were polished with 1-µm to 5-µm diamond paste. For e.max bars, the bars were machined to the final dimension because no compensation for shrinkage during sintering was required. The dimension of the samples was measured with a Vernier caliper with digital readout (Mitutoyo Corp., Tokyo, Japan), and the depth of the notches was measured with a measuroscope (Nikon UM-2 measuroscope, Japan) (Figure 3) by taking the average depth of the notch ends on both side (a1 and a2). All the bars were polished with 240-grit, 320-grit, 400-grit, 600-grit paper. The flexural strength bars were beveled at all line angles. All zirconia bars were sintered in Thermo Scientific™ Lindberg/Blue M 1700°C furnace (Waltham, MA) following the heating schedule specified by each company (Table III). IPS e.max bars were sintered in the Programat CS furnace.

FLEXURAL STRENGTH AND MODULUS

In testing the bar shape samples, a three-point bending test was used to measure the uniaxial flexural strength (F) and flexural modulus (E) on a universal testing machine (MTS Sintech ReNew 1123, MTS Systems Corporation, St. Paul, MN) (Figure 4). The following formula was be used:

$$F = \frac{3P_f L}{2BH^2}$$

$$E = \frac{3P_f L^3}{4DBH^3}$$

Where, P_f is the measured load at fracture, L the length, B the width, H the height of the specimen, D the deflection due to the load applied at the middle of the beam. The loading rate of the cross head was be 0.5mm/min at room temperature (25±1 °C),

FRACTURE TOUGHNESS

The single edge V-notch beam (SEVNB) method in three point flexure mode was used to measure the fracture toughness by the universal testing machine. The loading rate of the cross head was be 0.5mm/min at room temperature (25±1 °C).The following equations [56] were used determine the value of fracture toughness:

$$K_{IC} = \frac{P_f LY}{BH^{1.5}}$$

$$Y = \frac{3(a/H)^{0.5}\ [1.99 - a/H(1 - a/H)(2.15 - 3.93a/H + 2.7(a/H)^2)]}{2(1 + 2(a/H))(1 - a/H)^{1.5}}$$

And where a is the notch depth.

CROWN PREPARATION, DESIGN, AND FABRICATION

A preparation for zirconia crowns was prepared on a dentform right mandibular first molar with round shoulder finishing line using a 1-mm thick rounded shoulder diamond bur.[57-58] The occlusal and lateral reductions for zirconia material followed the ideal reduction specified by the companies, which is at least 1 mm (Figure 5). The IPS e.max preparation on another dentform right mandibular first molar followed the protocol of a previous study[59] and as recommended by Ivoclar, USA, with at least a 1.5-mm axial and 2-mm occlusal reduction and a rounded shoulder finishing line (Figure 5). A plastic angle guide sheet was used to assure 8-degree to 10-degree divergent walls (Figure 6).

An impression was taken before preparing the teeth with polyvinyl siloxane impression material (Exaflex, GC America) and was placed back on the prepared teeth to ensure the desired axial and occlusal reductions were obtained.[60-61] In addition, the preps were checked by two IUSD faculty. The two prepared teeth were scanned with a CAD/CAM machine (E4D Dentist, Texas, USA) using DentalogicTM 4.5 software. An unprepared tooth was scanned as well to act as a clone for zirconia and IPS e.max final designs. The design of IPS e.max was made, checked for the desired thickness (Figure 7A), and sent to a milling unit (E4D Dentist) located in University of Indiana School of Dentistry to make IPS e.max crowns (Figure 8). In the same procedure, the design of zirconia was made, checked for the desired thickness (Figure 7B), and sent to another milling machine (Roland DWX-50, California) located at a local lab in Indiana to make the zirconia crowns (Figure 9). For both designs, the cement space was set to 0.1 mm and the margin ramp to 0.25 mm. The material blocks and disks were ordered from manufacturers or authorized dental labs, and were milled by the milling machines to the desired crown design. IPS e.max crowns were sintered in Programat CS furnace (Ivoclar Vivadent, Ontario, Canada) (Figure 10A) following the manufacturers' instruction (Table III) without glazing. Zirconia crowns were sintered with Sintra furnace (Shenpaz, Dental, Israel) (Figure 10B) according to the manufacturers' instruction (Table III) without glazing.

DIE PREPARATION

Six polyvinyl siloxane impressions (Examix NDS, GC America) were taken (one for each group) (Figure 6B,D), and poured 8 times with a die material to make a total of 48 dies. Epoxy resin (EpoKwick® Epoxy System, Buehler, IL) was chosen for die material due to their similar flexural modulus to dentin.[62]

To have a standard vertical axis for all dies that matches the vertical axis of the prepared dentform (master die), box shape bases were made out of epoxy resin, EpoKwick® Epoxy System, to act as bases for the dies, and the top of each base was prepared to receive a die. A crown was seated on the prepared dentform tooth (master die), then the tooth was secured in a holding jig (Figure 11). The jig was placed in a universal testing machine, and adjusted so that the vertical axis of the tooth with the crown is angulated 100° to the vertical axis of loader. The loader was lower down until lightly contacting the crown in the central fossa. To keep the 10° angle, the crown was attached to the loader with a sticky wax, and then the loader was lifted along with the crown to be separated from the tooth. Each die was then seated on the attached crown, and a base was placed on the jig just below the attached crown and die. Superglue was applied on the top of the base, and then the loader was lowered down along with the attached crown and die until it touches the superglue-covered base. Ten minutes was allowed for the superglue to set in each die. The process was repeated for all dies.

SURFACE TREATMENT

Before bonding, the internal surfaces of the zirconia crowns were treated by air-abrasion (SandStorm, Vaniman, 10 50n) (Figure 12) with 50-µm aluminum oxide particles at 1 bar and a distance of 10 mm for 10 seconds.[62] IPS e.max CAD crowns were etched with 5.0-percent hydrofluoric acid (IPS Ceramic etching gel, Ivoclar Vivadent) for 20 seconds according to the manufacturer's instruction. All the crowns were cleaned in an ultrasonic cleaner for 5 minutes, air dried, and silanized with ESPE Sil, 3M ESPE.[64] Five minutes were allowed to elapse for silane reaction.[64] No outer surface treatment was done for any of the crowns.

CEMENTATION

Before cementation, few crowns were selected randomly for marginal fitting and were evaluated by one evaluator (Figure 13). RelyX™ Unicem resin cement was used to cement all the crowns without light curing, following manufacturer's instructions,[63,65,66] which is also recommended by all zirconia manufacturing laboratories. All the crowns were cemented to epoxy dies without any surface modification to the die as described by Yucel et al.[62]

During cementation, the crowns were seated with finger pressure to ensure proper cementation. Excess cement was removed 2 minutes after seating by an explorer, and continued pressing for additional 6 minutes.

LOAD TO FAILURE TESTING

A universal machine (Instron E3000, Norwood, MA) (Figure 14) was used to perform load to fracture test after 14 hours of cementation. The loader is an 11-mm diameter custom made stainless steel rod with 4-mm diameter rounded end. Before loading the restorations, a 1-mm thickness aluminum pad was used as a stress breaker between the crown and the loader, and to minimize surface damage.[67,68] All restorations were loaded at 10° to the long axis of the tooth at 1 mm/min until fracture (Figure 15). The data were recorded to compare the load needed to fracture the crowns.

SEM

Both unsintered specimens and fractured pieces of the tested crowns were gold-plated and imaged under scanning electron microscope (SEM) (JEOL 6390 LV, Jeol USA, Peabody, MA).

STATISTICAL METHODS

One-way ANOVA followed by pair-wise comparisons was used to evaluate the effects of group (Bruxzir, e.max, FZ, KDZ, QZ, and Suntech) on flexural strength, modulus, fracture toughness, and fracture resistance. In addition, the Weibull characteristic strength and modulus parameters were estimated using survival analysis. The pair-wise comparisons were also provided based on Weibull survival analysis. The significance level was set at 5 percent.

SAMPLE SIZE JUSTIFICATION

Based on prior studies the within-group standard deviations for fracture strength, flexural strength, and fracture toughness are estimated to be 200 N, 50 MPa, and 0.5 $MPa/m^{1/2}$, respectively. With a sample size of 6 specimens per group, the study will have

80-percent power to detect differences between any two groups of 90 MPa for flexural strength, and 0.89 MPa/m$^{1/2}$ for fracture toughness, assuming two-sided tests each conducted at a 5-percent significance level. In addition, with a sample size of 8 specimens per group, the study will have 80-percent power to detect differences between any two groups of 301 N for fracture strength under the same assumption.

RESULTS

RESULTS FROM SEM OF UNSINTERED MATERIALS

Under SEM, all CAD/CAM zirconia materials show highly porous structures. The particles as well as the pores are in the sub-micrometer range. (Figure 16). The porous structure is expected to provide an appropriate mechanical property allowing the milling process to take place. The porous structure also explains the large volumetric shrinkage after sintering (Table II).

RESULTS FROM BAR SAMPLES

One specimen from ST group was excluded because it was defective during sintering. The flexural strength, modulus, fracture toughness, fracture resistance values are listed in Table IV, Table V.

In flexural strength, QZ shows the highest value of 788.12 (44.51) MPa while e.max shows the lowest value of 325.87 (20.4) (Table IV) (Figure 17). The non-translucent QZ is significantly higher than all the translucent zirconia materials.

In fracture toughness, QZ also shows the highest value of 6.85 (1.27) MPa $m^{1/2}$ while e.max shows the lowest value of 3.29 (0.46) (Table IV) (Figure 18).

In flexural modulus, QZ, KDZ, and FZ show no differences while e.max show the lowest value of 62.53 (3.51) GPa (Table IV) (Figure 19).

RESULT FROM CROWNS

In crown fracture resistance, QZ shows the highest value of 2489.8 (165.49) N with no statistical difference with any of the other groups, except Suntech, which shows a significantly lower value of 2131.8 (153.2) N ($p < 0.05$) (Table V)(Figure 20).

Survival probability (Kaplan-Meier) of flexural strength, fracture toughness, and fracture resistance for the groups are shown in Figure 21 through Figure 23.

In summary, e.max has significantly lower flexural strength and fracture toughness than all zirconia brands. There was no significant difference between fracture resistance of e.max and other groups. QZ showed significantly higher flexural strength than other zirconia groups (Table VI to Table IX).

WEIBULL PARAMETERS ANALYSIS

Table IX shows the Weibull Characteristic and modulus along with SE and confidence interval. The Weibull Characteristic number corresponds to the fracture toughness, flexural strength, and fracture resistance level for a 63.2-percent probability of failure. The Weibull modulus reflects the extent of data variability. Higher Weibull modulus indicates smaller data variability among samples. In both fracture toughness and flexural strength, FZ group show the highest Weibull modulus, or the smallest scattering in data. QZ has the highest average fracture toughness among all groups, but shows the lowest Weibull modulus, indicating a larger scattering or wider distribution of data.

RESULTS FROM SEM

The SEM (FIGURE 24) showed a circular crack on the occlusal surface that was seen in most of the crowns. This circular fracture in all crowns is cone-shaped with the base toward the die.

TABLES AND FIGURES

TABLE I

The materials used in this study

Group	Brands	Manufacturers	Materials
1	BruxZir (BZ)	Glidewell Dental labs, Newport Beach, CA, USA	Translucent Zirconia
2	KDZ Bruxer (KZ)	Keating Dental Arts, Irvine, CA, USA	Translucent Zirconia
3	Suntech (ST)	Sun Dental Labs, Clearwater, Florida, USA	Translucent Zirconia
4	CAP FZ (FZ)	Custom Automated Prosthetics, Stoneham, MA, USA	Translucent Zirconia
5	CAP QZ (QZ) Control	Custom Automated Prosthetics, Stoneham, MA, USA	Traditional, non-translucent Zirconia
6	IPS e.maxCAD (EX) Control	Ivoclar Vivadent, Liechtenstein, Germany	Lithium disilicate

TABLE II

Shrinkage factor and oversize cutting percentage*

Brands	Shrinkage factor	Oversize cutting percentage
BruxZir	1.23	23%
KDZ Bruxer	1.243	24.3%
Suntech	1.25	25%
CAP FZ	1.25	25%
CAP QZ	1.2458	24.58%
IPS e.max CAD	N/A	N/A

*Compensates for shrinkage after sintering.

TABLE III

The sintering cycle in C° for all samples

FZ CAP	
Temp 1	25
Rate	8 C /min
Temp 2	980
Hold	1 min
Rate	6 C/min
Temp 3	1550
Hold	2 hrs
Temp 4	1550
Cooling time	1.5 hr
Temp 5	400
Free cooling	To 25 C

Suntech	
Temp 1	20
Rate	2:00 hr
Temp 2	990
Rate	1:15 hr
Temp 3	1600
Hold	2:00 hr
Temp 4	1600
Cool time	3:15
Temp 5	25

IPS e.max CAD	
Stand by temp	403
Closing time mm:ss	6:00
Temp increase	90/30
Holding temp in	820/840
Holding time mm:ss	00:10/7:00
Vacuum on temp	550/820
Vacuum off temp	820/840
long-term cooling	700

QZ CAP	
Temp 1	25
Rate	20 C /min
Temp 2	980
Hold	1 min
Rate	10 C/min
Temp 3	1530
Hold	2 hrs
Temp 4	1530
Cooling time	1.5 hr
Temp 5	400
Free cooling	To 25

KDZ	
Temp 1	25
Rate	5 C/min
Temp 2	1000
Rate	2 C/min
Temp 3	1590
Hold	3 hrs
Temp 4	1590
Cool rate	3-5 C/min
Temp 5	25

BruxZir	
Temp 1	25
Rate	10 C/min
Temp 2	1530
Hold	2 hrs
Temp 3	1530
Cool rate	4 C/min
Temp 4	25

TABLE IV

The mean, standard deviation, standard error, minimum and maximum for flexural strength (MPa), fracture toughness ($MPa.m^{1/2}$), modulus (GPa)*

Outcome	Group	N	Mean	Standard Deviation	Standard Error	Minimum	Maximum
Flexural Strength	e.max	6	325.87^{e}	20.4	8.33	304.71	359.4
	BruxZir	6	558.6^{d}	49.18	20.08	500.58	620.77
	FZ	6	714.61^{b}	32.98	13.46	656.33	747.19
	KDZ	6	613.62^{c}	33.83	13.81	565.92	654.16
	QZ	6	788.12^{a}	44.51	18.17	719.54	840.11
	Suntech	6	622.85^{c}	54.05	22.07	563.25	684.93
Fracture Toughness	e.max	6	3.29^{c}	0.46	0.19	2.69	3.88
	BruxZir	6	6.45^{a}	0.87	0.35	5.25	7.13
	FZ	6	$6.35^{a,b}$	0.29	0.12	6.02	6.74
	KDZ	6	$6.09^{a,b}$	0.87	0.35	4.93	7.23
	QZ	6	6.85^{a}	1.27	0.52	5.8	9.08
	Suntech	6	5.42^{b}	0.79	0.32	4.27	6.65
Modulus	e.max	6	62.528^{d}	3.5175	1.436	58.855	68.481
	BruxZir	6	76.404^{c}	3.2484	1.3262	71.619	80.241
	FZ	6	$97.359^{a,b}$	7.3301	2.9925	83.562	103.913
	KDZ	6	104.801^{a}	3.8176	1.5585	101.281	111.022
	QZ	6	104.675^{a}	5.7023	2.3279	94.897	112.653
	Suntech	6	94.589^{b}	10.596	4.326	76.837	103.877

*Same letters indicate no statistical difference.

TABLE V

The mean, standard deviation, standard error, minimum and maximum for fracture resistance*

Outcome	Group	N	Mean	Standard Deviation	Standard Error	Minimum	Maximum
Fracture Resistance	e.max	8	2366.9[a,b]	262.02	92.64	2010.7	2778.8
	BruxZir	8	2382.3[a,b]	323.85	114.5	1825.7	2808.1
	FZ	8	2456.4[a,b]	211.78	74.88	2127.5	2694.9
	KDZ	8	2232.9[a,b]	380.34	134.47	1538.4	2875.2
	QZ	8	2489.8[a]	165.49	58.51	2187.5	2641.3
	Suntech	7	2131.8[b]	153.2	57.91	1951.4	2350.8

*Same letters indicate no statistical difference.

TABLE VI

Pair-wise ANOVA and Weibull for flexural strength

Outcome	Comparison	Difference	P-value	P-value Weibull Survival	Comparison Weibull Survival
Flexural Strength	BruxZir > e.max	232.72	<.0001	<0.0001	BruxZir > e.max
	BruxZir < FZ	-156	<.0001	<0.0001	BruxZir < FZ
	BruxZir < KDZ	-55.02	0.0262	0.02	BruxZir < KDZ
	BruxZir < QZ	-229.5	<.0001	<0.0001	BruxZir < QZ
	BruxZir < Suntech	-64.26	0.0105	0.009	BruxZir < Suntech
	e.max < FZ	-388.7	<.0001	<0.0001	e.max < FZ
	e.max < KDZ	-287.7	<.0001	<0.0001	e.max < KDZ
	e.max < QZ	-462.3	<.0001	<0.0001	e.max < QZ
	e.max < Suntech	-297	<.0001	<0.0001	e.max < Suntech
	FZ > KDZ	101	0.0002	<0.0001	FZ > KDZ
	FZ < QZ	-73.51	0.0039	<0.0001	FZ < QZ
	FZ > Suntech	91.76	0.0005	0.0002	FZ > Suntech
	KDZ < QZ	-174.5	<.0001	<0.0001	KDZ < QZ
	KDZ and Suntech n.s.	-9.23	0.6976	0.421	KDZ & Suntech n.s.
	QZ > Suntech	165.27	<.0001	<0.0001	QZ > Suntech

TABLE VII

Pair-wise ANOVA and Weibull for fracture resistance

Outcome	Comparison	Difference	P-value	P-value Weibull Survival	Comparison Weibull Survival
Fracture Resistance	BruxZir & e.max n.s.	15.5	0.9074	0.806	BruxZir & e.max n.s.
	BruxZir & FZ n.s.	-74.05	0.5789	0.787	BruxZir & FZ n.s.
	BruxZir & KDZ n.s.	149.41	0.2656	0.423	BruxZir & KDZ n.s.
	BruxZir & QZ n.s.	-107.4	0.4217	0.683	BruxZir & QZ n.s.
	BruxZir & Suntech n.s.	250.52	0.0748	0.0035	BruxZir > Suntech
	e.max & FZ n.s.	-89.55	0.5025	0.557	e.max & FZ n.s.
	e.max & KDZ n.s.	133.91	0.3177	0.537	e.max & KDZ n.s.
	e.max & QZ n.s.	-122.9	0.3585	0.447	e.max & QZ n.s.
	e.max & Suntech n.s.	235.03	0.0938	0.0058	e.max > Suntech
	FZ & KDZ n.s.	223.46	0.099	0.263	FZ & KDZ n.s.
	FZ & QZ n.s.	-33.39	0.8021	0.861	FZ & QZ n.s.
	FZ > Suntech	324.57	0.0226	<0.0001	FZ > Suntech
	KDZ & QZ n.s.	-256.8	0.0592	0.209	KDZ & QZ n.s.
	KDZ & Suntech n.s.	101.11	0.4648	0.171	KDZ & Suntech n.s.
	QZ > Suntech	357.96	0.0125	<0.0001	QZ > Suntech

TABLE VIII

Pair-wise ANOVA and Weibull for fracture toughness

Outcome	Comparison	Difference	P-value	P- value Weibull Survival	Comparison Weibull Survival
Fracture Toughness	BruxZir > e.max	3.16	<.0001	<0.0001	BruxZir > e.max
	BruxZir & FZ n.s.	0.1	0.8353	0.263	BruxZir & FZ n.s.
	BruxZir & KDZ n.s.	0.36	0.4571	0.412	BruxZir & KDZ n.s.
	BruxZir & QZ n.s.	-0.41	0.3974	0.319	BruxZir & QZ n.s.
	BruxZir > Suntech	1.02	0.0384	0.011	BruxZir > Suntech
	e.max < FZ	-3.06	<.0001	<0.0001	e.max < FZ
	e.max < KDZ	-2.8	<.0001	<0.0001	e.max < KDZ
	e.max < QZ	-3.57	<.0001	<0.0001	e.max < QZ
	e.max < Suntech	-2.14	<.0001	<0.0001	e.max < Suntech
	FZ & KDZ n.s.	0.26	0.5907	0.921	FZ & KDZ n.s.
	FZ & QZ n.s.	-0.51	0.2939	0.091	FZ & QZ n.s.
	FZ & Suntech n.s.	0.92	0.0599	0.031	FZ > Suntech
	KDZ & QZ n.s.	-0.76	0.1174	0.134	KDZ & QZ n.s.
	KDZ & Suntech n.s.	0.67	0.1682	0.114	KDZ & Suntech n.s.
	QZ > Suntech	1.43	0.0051	0.006	QZ > Suntech

TABLE IX

Pair-wise ANOVA for modulus

Outcome	Comparison	Difference	P-value
Modulus	BruxZir > e.max	13876	0.0006
	BruxZir < FZ	-20955	<.0001
	BruxZir < KDZ	-28397	<.0001
	BruxZir < QZ	-28270	<.0001
	BruxZir < Suntech	-18185	<.0001
	e.max < FZ	-34831	<.0001
	e.max < KDZ	-42272	<.0001
	e.max < QZ	-42146	<.0001
	e.max < Suntech	-32061	<.0001
	FZ < KDZ	-7442	0.0487
	FZ & QZ n.s.	-7316	0.0524
	FZ & Suntech n.s.	2769.5	0.4504
	KDZ & QZ n.s.	126.07	0.9725
	KDZ > Suntech	10211	0.0084
	QZ > Suntech	10085	0.0092

TABLE X

Weibull parameter for flexural strength,
fracture toughness, fracture resistance

Outcome	*Group*	*Weibull Characteristic*	*SE for Weibull Characteristic*	*95% CI for Weibull Characteristic*	*Weibull Modulus*	*SE for Weibull Modulus*	*95% CI for Weibull Modulus*
Fracture Toughness	BruxZir	6.8[a]	0.255	(6.3, 7.3)	11.4	4.2	(3.2, 19.6)
	e.max	3.4[c]	0.166	(3.8, 3.1)	9.1	3.0	(3.3, 14.9)
	FZ	6.5[a]	0.109	(6.3, 6.7)	25.9	8.2	(9.8, 41.9)
	KDZ	6.4[ab]	0.323	(5.8, 7.1)	8.7	2.8	(3.2, 14.1)
	QZ	7.4[a]	0.546	(6.3, 8.4)	5.9	1.8	(2.4, 9.3)
	Suntech	5.7[b]	0.307	(5.1, 6.3)	8.1	2.5	(3.2, 13.0)
Flexural Strength	BruxZir	579.2[d]	17.2	(545.5, 612.9)	14.6	4.8	(5.2, 23.9)
	e.max	335.1[e]	8.5	(318.5, 351.7)	17.2	5.2	(7.1, 27.4)
	FZ	727.8[b]	9.5	(709.1, 746.5)	32.9	11.3	(10.8, 54.9)
	KDZ	628.1[c]	11.4	(605.7, 650.5)	23.7	7.8	(8.4, 39.1)
	QZ	807.0[a]	15.1	(777.5, 836.5)	23.1	7.6	(8.3, 38.0)
	Suntech	685.2[c]	33.8	(618.9, 751.5)	14.6	4.8	(5.2, 24.1)
Fracture Resistance	BruxZir	2512.7[a]	98.85227	(2319.0, 2706.5)	9.5	2.7	(4.2, 14.8)
	e.max	2480.0[a]	65.86629	(2350.9, 2609.1)	10.3	2.8	(4.8, 15.7)
	FZ	2544.1[a]	59.77631	(2426.9, 2661.3)	15.9	4.7	(6.7, 25.1)
	KDZ	2383.3[a,b]	128.1165	(2132.2, 2634.4)	7.0	1.8	(3.3, 10.6)
	QZ	2556.9[a]	42.10976	(2474.4, 2639.4)	22.6	6.9	(9.0, 36.2)
	Suntech	2198.6[a]	52.6498	(2095.4, 2301.8)	16.8	5.0	(7.1, 26.6)

FIGURE 1. Isomet 1000, a cutting machine.

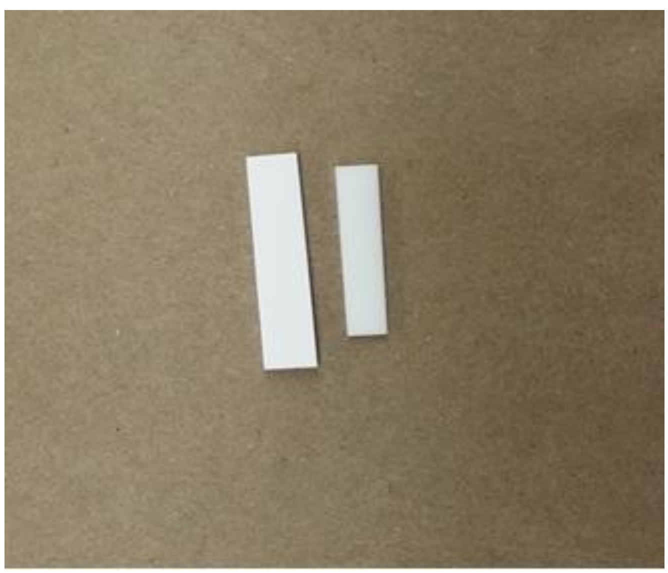


FIGURE 2. Zirconia bars, pre-sintered on left, and sintered on right.

FIGURE 3. Nikon measuroscope used to measure and examine the notches.

FIGURE 4. MTS Sintech 123, a loading machine.

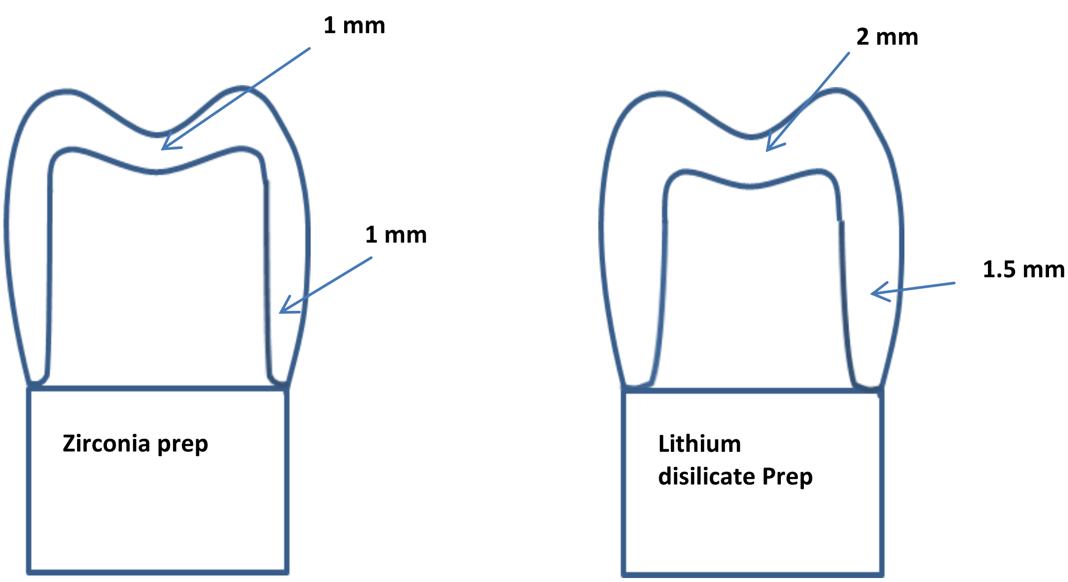


FIGURE 5. Preparation designs for zirconia and IPS e.max crowns.

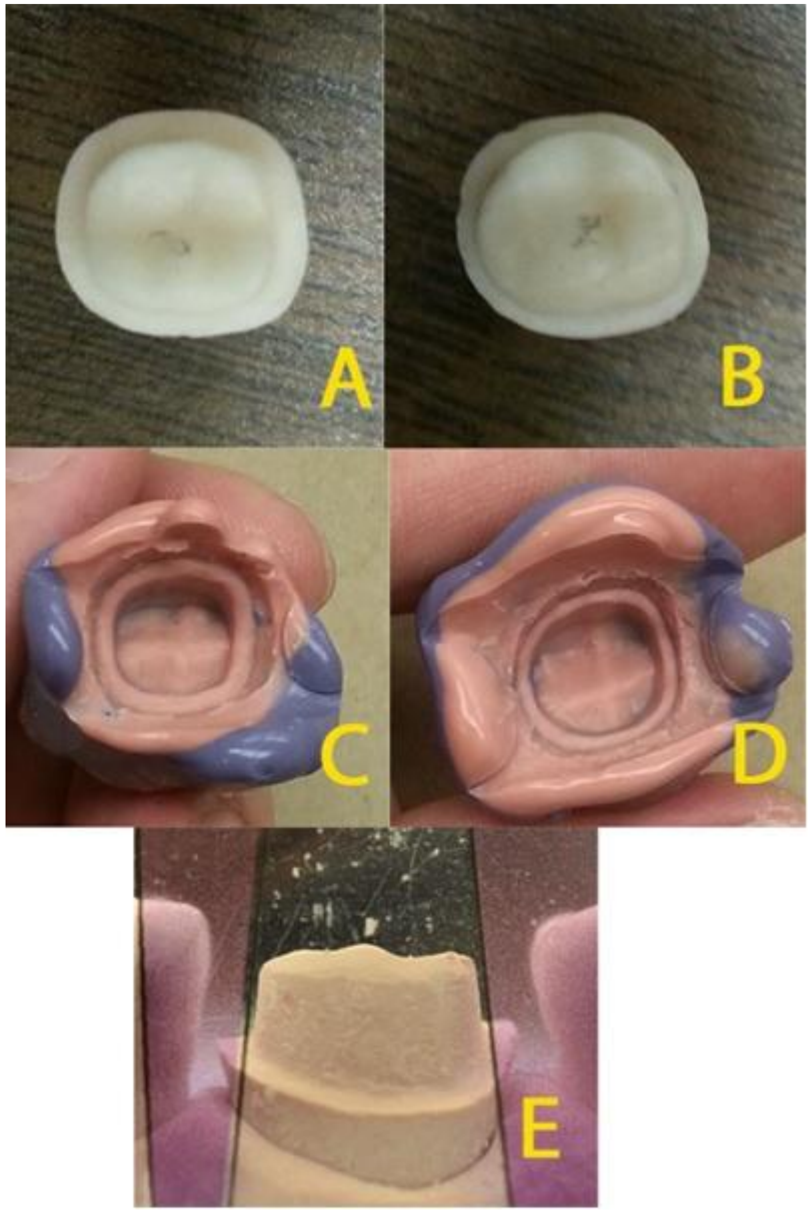


FIGURE 6. The master die for e.max. A. The master die for zirconia. B. The impression of e.max master die; C.The impression of zirconia master die; D and E. Checking a 10-degree axial angulation of prep with an angulation sheet guide.

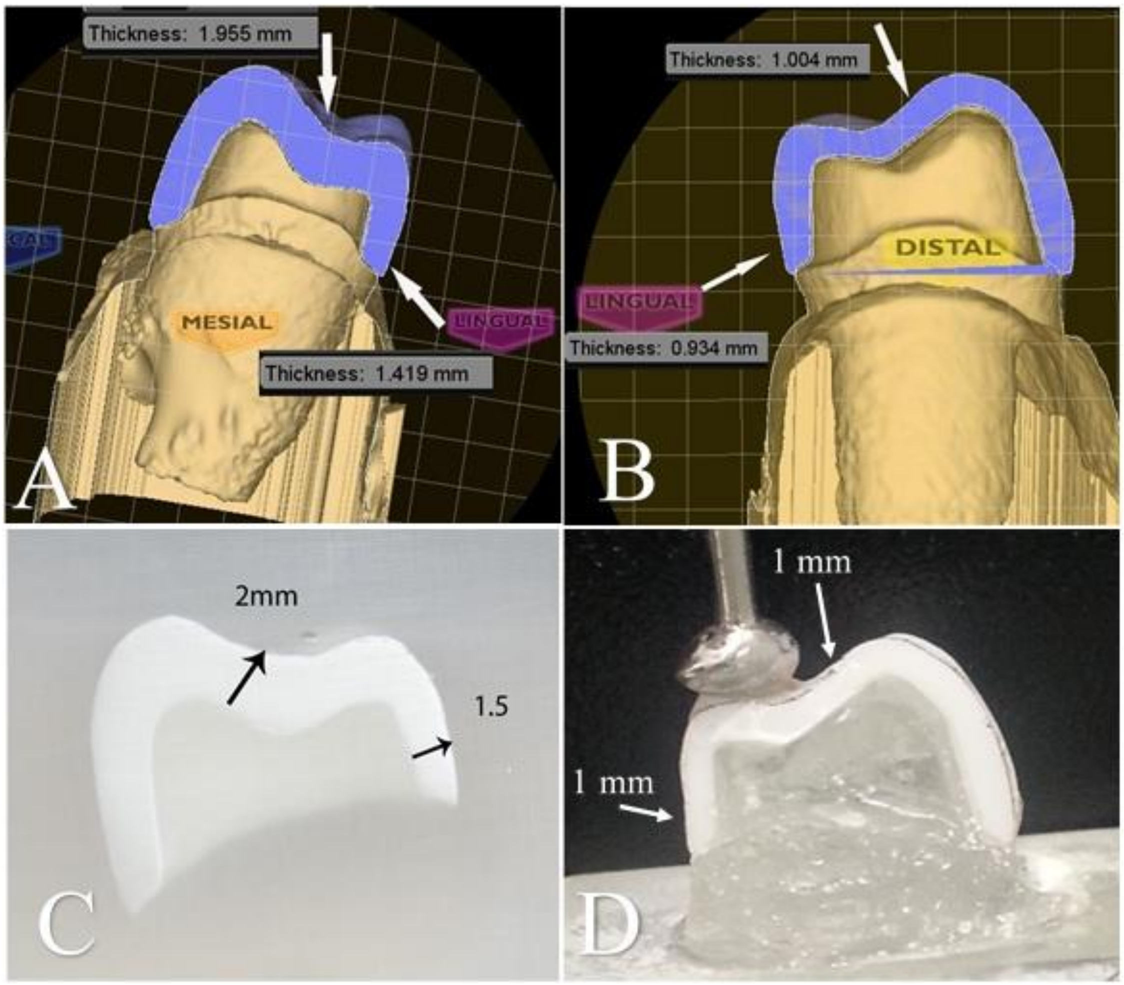


FIGURE 7. E.max design. A. Zirconia design; B. Milled e.max crown, and C. Zirconia milled crown.

FIGURE 8. ED4D CAD/CAM milling machine for e.max.

FIGURE 9. Roland CAD/CAM machine for zirconia.

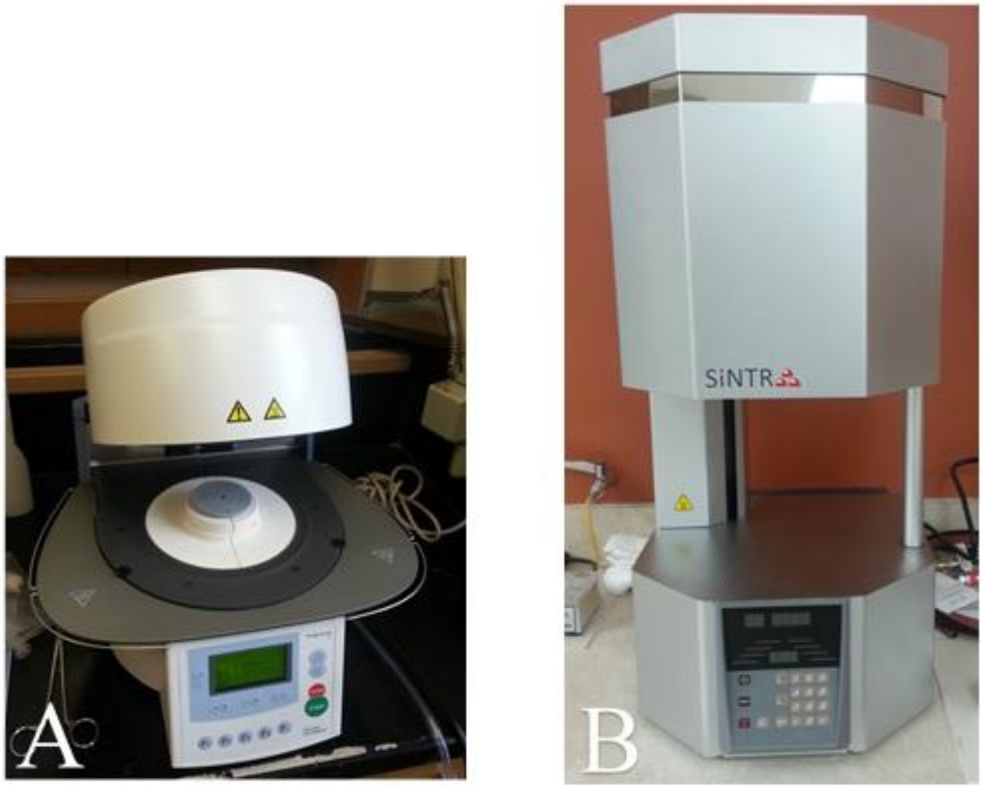


FIGURE 10.
A. Programmat S1, for e.max sintering.
B. Sintra, for zirconia sintering.

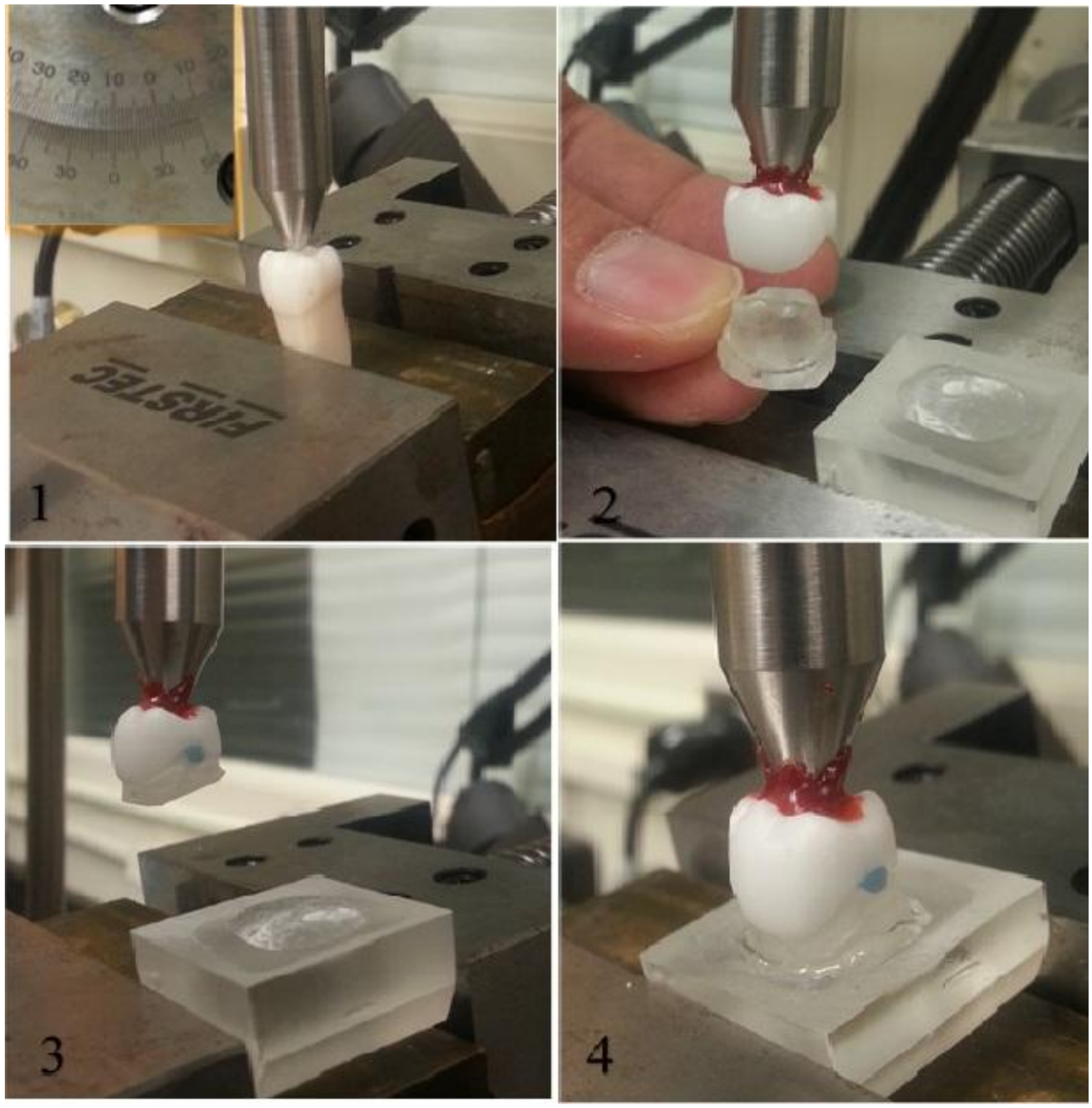


FIGURE 11. Setting the dies at 10° angle.

FIGURE 12.   Sandstorm sandblasting device.

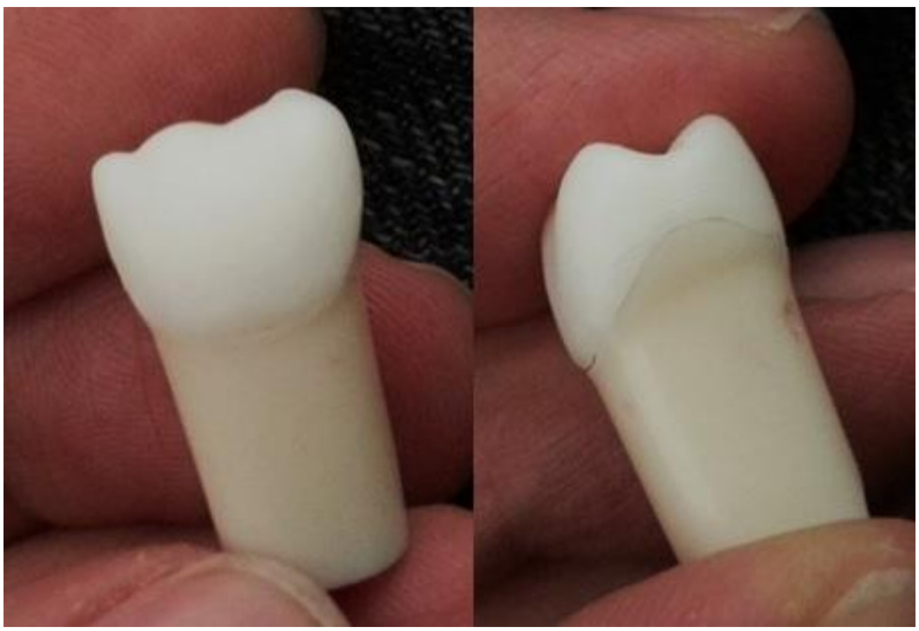


FIGURE 13. Examination of crowns for marginal fitting on the master die.

FIGURE 14. Instron E3000, loading machine.

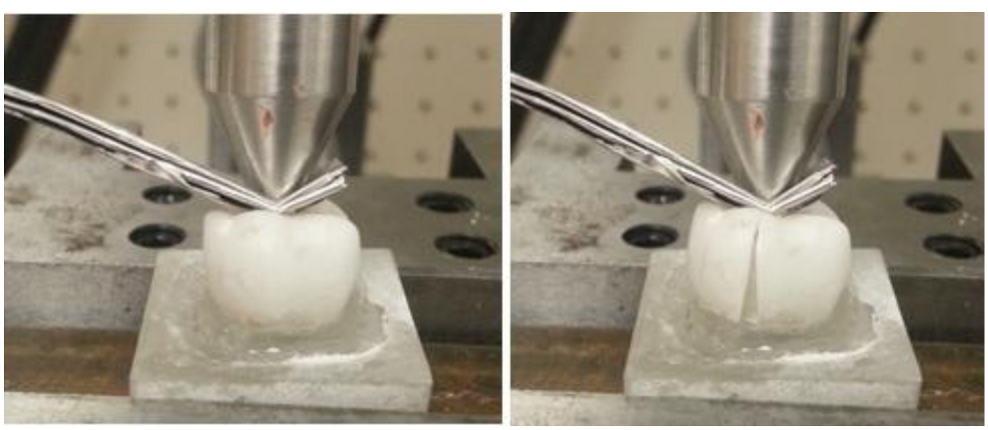
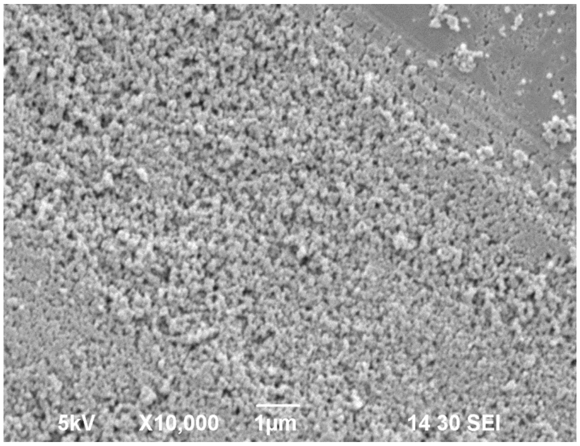
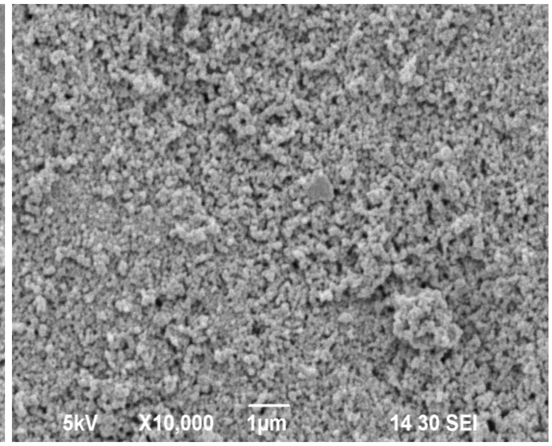


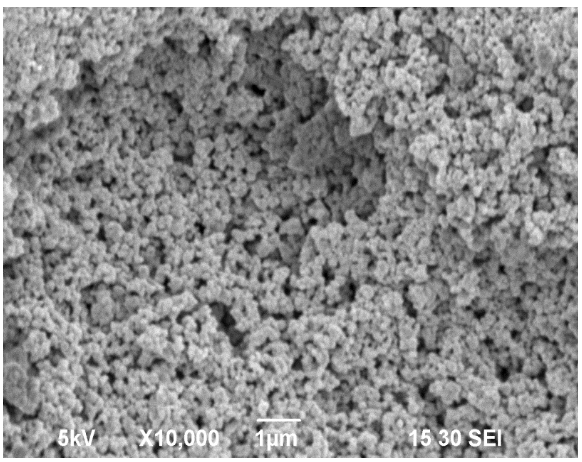
FIGURE 15. A crown under loading and when fractured.



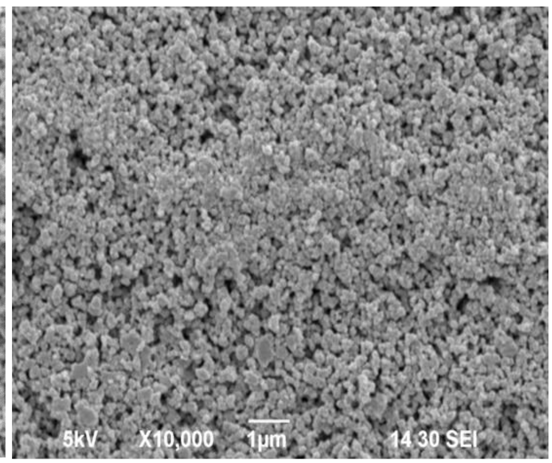
FZ-unsintered-10k-1



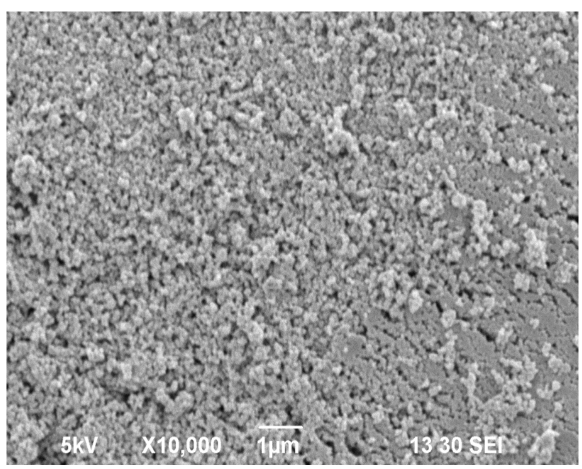
KZ-unsintered-10k-1



QZ-unsintered-10k-1



BX-unsintered-10k-1



ST-unsintered-10k

FIGURE 16. Nanoparticles of FZ, KZ, QZ, BX, and ST before sintering under SEM (X10000 magnification).

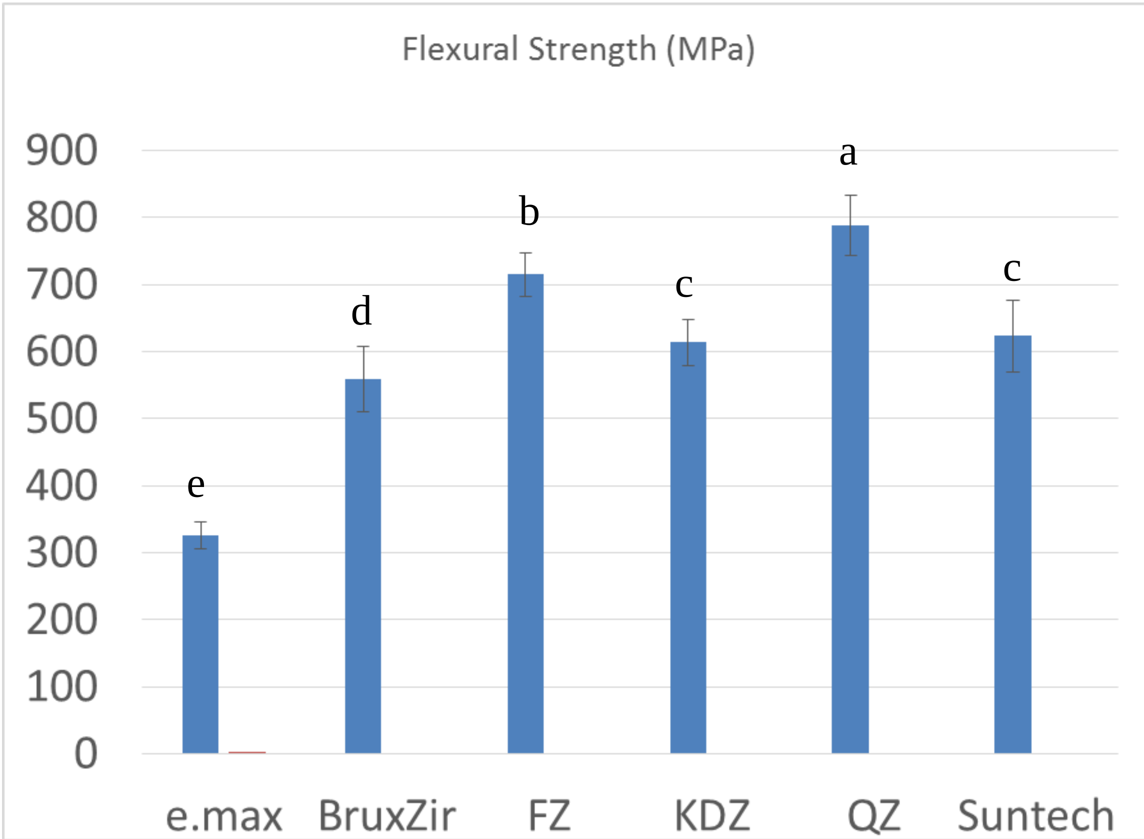


FIGURE 17. The mean flexural strength of all materials. Same letters indicate no statistical difference.

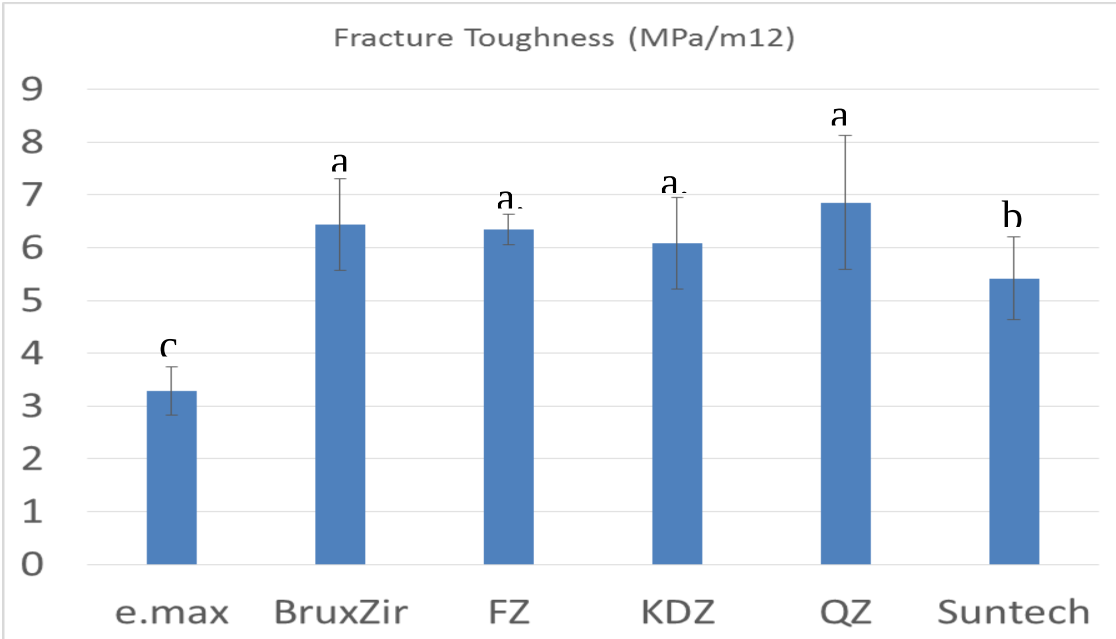


FIGURE 18. The mean fracture toughness of all materials. Same letters indicate no statistical difference.

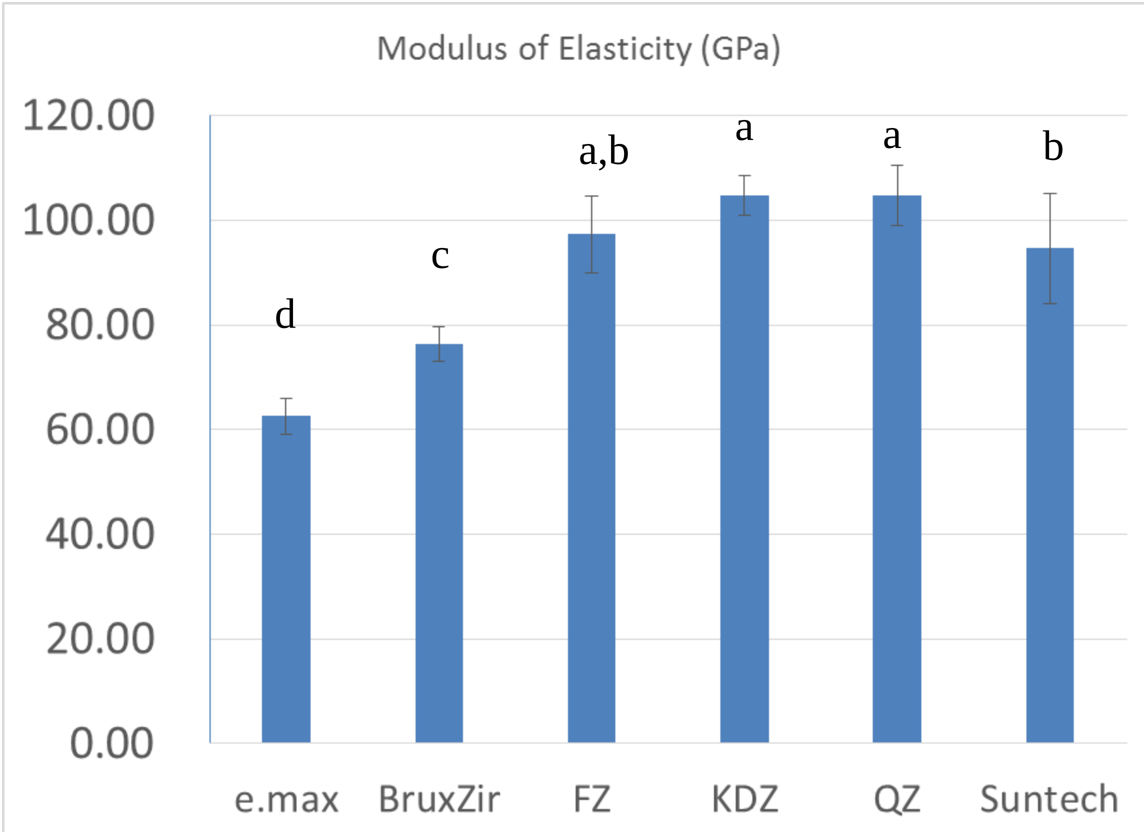


FIGURE 19. The mean elastic modulus of all materials. Same letters indicate no statistical difference.

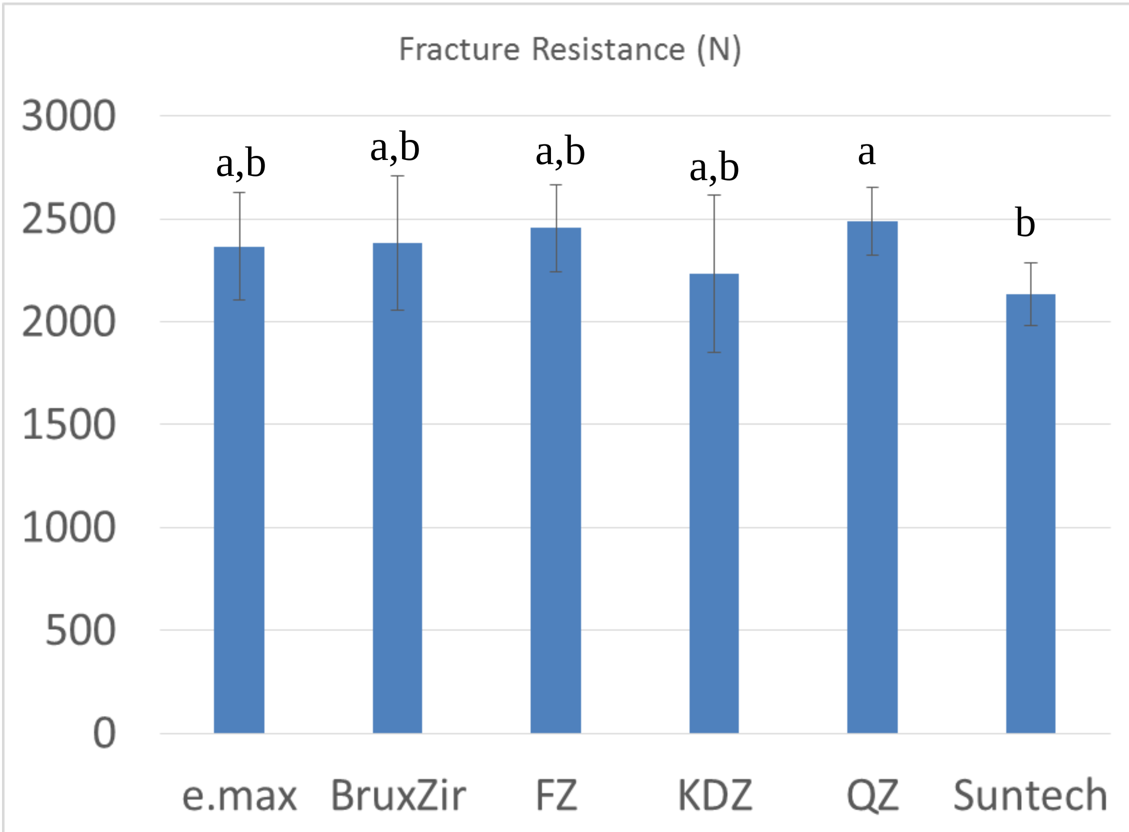


FIGURE 20. The mean fracture resistance of all materials. Same letters indicate no statistical difference.

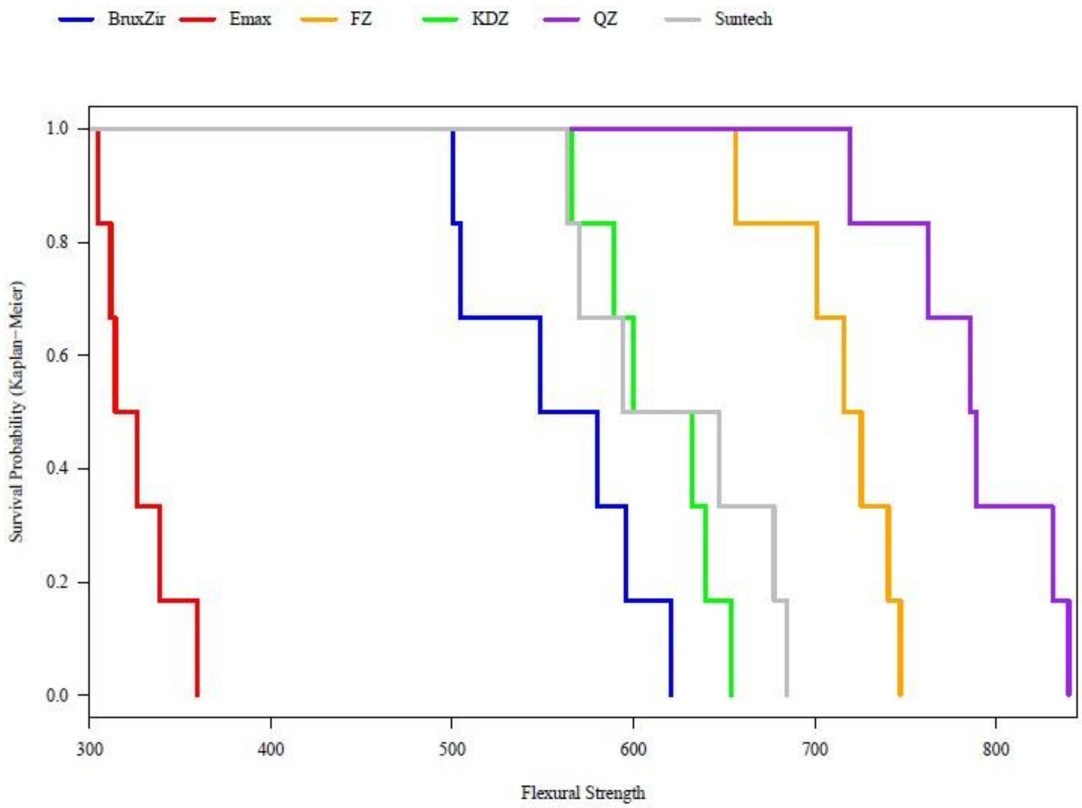


FIGURE 21. Survival probability (Kaplan-Meier) of flexural strength of the materials.

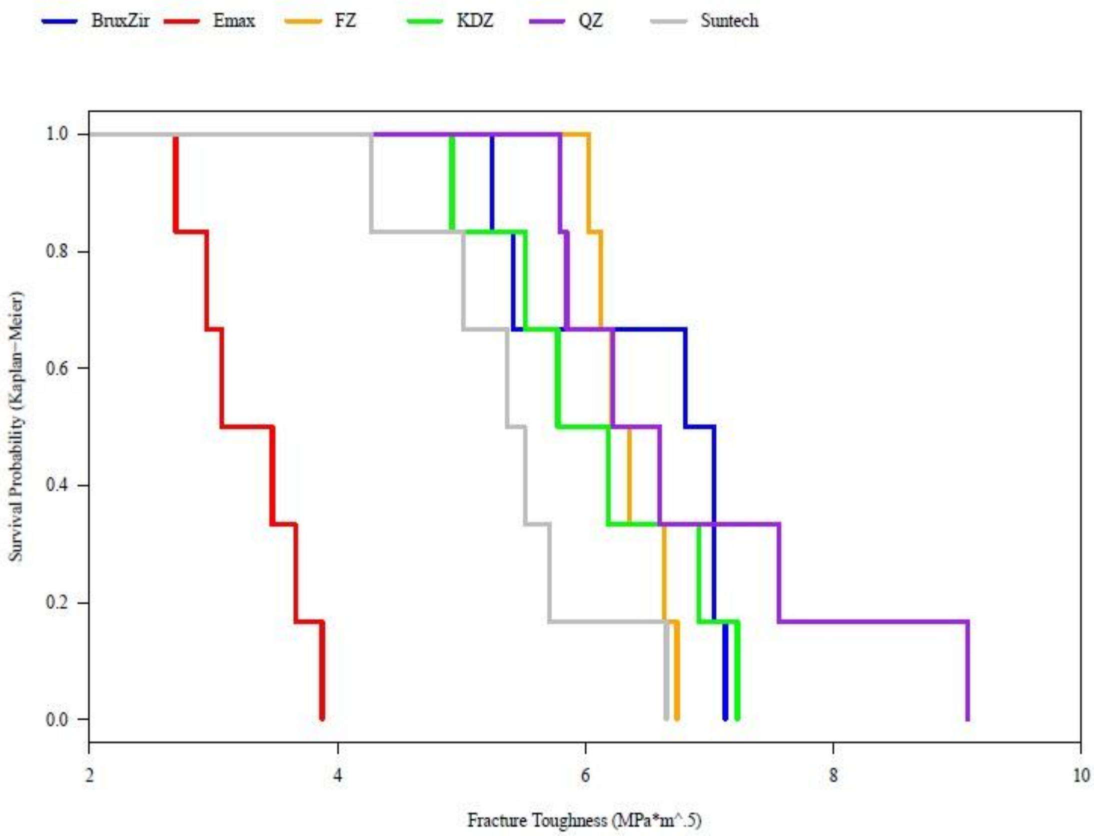


FIGURE 22. Survival probability (Kaplan-Meier) of fracture toughness of the materials.

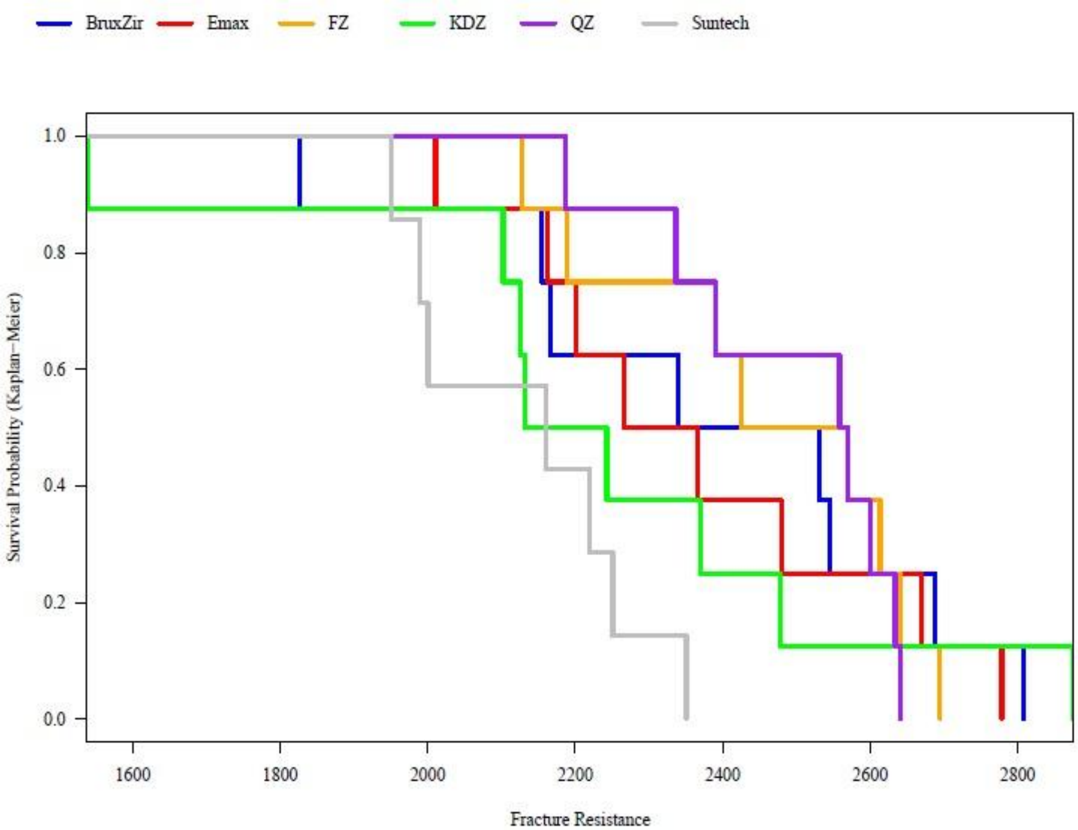
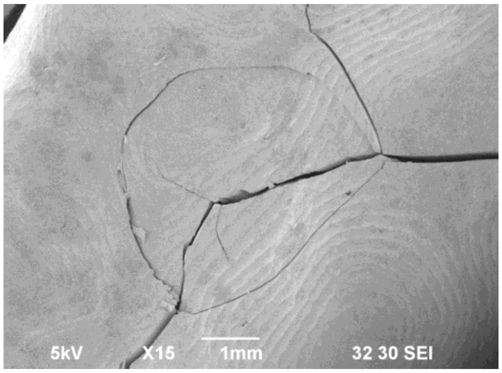
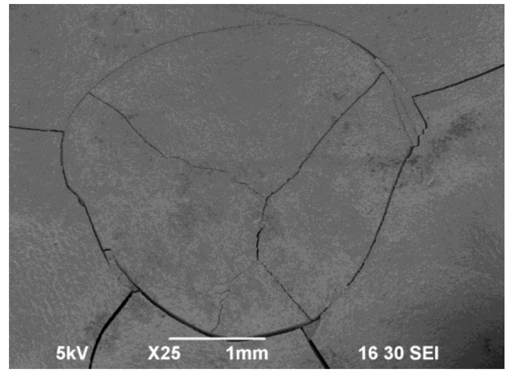


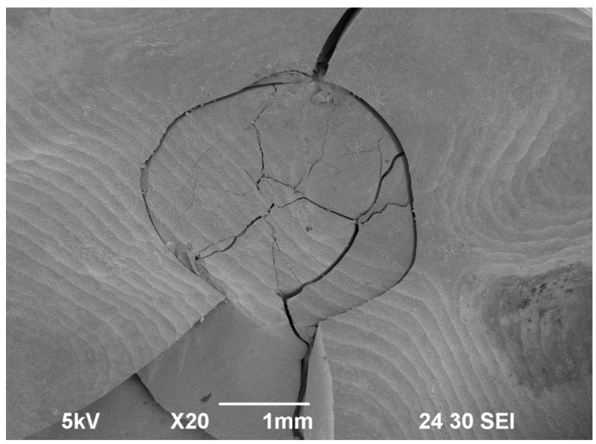
FIGURE 23. Survival probability (Kaplan-Meier) of fracture resistance of the materials.





BX15x_1

EM-crown-25x-1



FZ-crown-20x-1

FIGURE 24. Fracture pattern of BX, EX, FZ crown under SEM (X20 magnification).

DISCUSSION

Lithium disilicate crowns receive dentists' attention due to the material's relatively high strength, high esthetics, and promising clinical results. Currently, IPS e.max Press is the available lithium disilicate brand in the markets, which replaced the previous IPS Empress II. It is reported to have 400-MPa flexural strength[69]and is usually used for inlay, onlay, and crown substructure. A chair-side CAD/CAM version of IPS e.max has been revealed under the name "IPS e.max CAD" that can be milled to a monolithic crown. This would avoid the common problems associated with veneered restorations, such as chipping of the veneering layers,[4] which creates failures that cost lab time and money. The product was released recently, and not much information is available about the mechanical properties of IPS e.max CAD. Tysowsky reported the CAD/CAM version has a 360 MPa flexural strength, and 2.25 MPa.m$^{1/2}$ fracture toughness.[69]

In this study, the mechanical properties of IPS e.max CAD were investigated. The results showed 325.9 (20.4) MPa flexural strength and 3.3(0.5) MPa.m$^{1/2}$ fracture toughness, which are comparable to what Tysowsky found.

Zirconia is now being used as crown/bridge substructure instead of metal more often than before because of its biocompatibility and the ongoing improvement in strength. Zirconia is usually strengthened by a toughening mechanism, which involves including oxides in the structure to stabilize the tetragonal phase at room temperature. Under stress, the tetragonal phase converts into monoclinic accompanied by 4-percent volume expansion exerting compressive force on the tip of the crack to prevent its propagation.[10]

Vita In-Ceram Zirconia is made by adding 33-percent (12 mol%) CeO_2 partially stabilized zirconia to In-Ceram Alumina. It is essentially a mixture of alumina and zirconia. It is widely used nowadays for all-ceramic substructures. Chong et al. investigated In-Ceram Zirconia and reported its flexural strength to be 513(69) MPa, where the specimens were polished with up to 600-μm grit paper.[70] This was similar to Apholt's results, in which a 624(58) MPa flexural strength was obtained when the specimens were polished with up to 1200 grit.[71] Papanagiotou tested In-Ceram Zirconia flexural strength under various surface treatments. The results show air-abraded specimens had the highest value, 950.2(127) MPa, compared with polished, 844(132) MPa, and not treated, 814(161) MPa.[72] That study inferred surface treatment affects the strength of In-Ceram zirconia.

Compared with In-Ceram Zirconia, Y-TZP (Yttria stabilized zirconia) is widely used as well and preferred as the transformation toughening mechanism is more pronounced. Most studies reported higher flexural strength and fracture toughness of Y-TZP than In-Ceram Zirconia. Stawarczyk compared the flexural strength of many brands of non-translucent Y-TZP (Zeno, ZR, Ceramill ZI, Copran, InCoris, Cercon ZR, and Lava Zirkon) with In-Ceram Zirconia. The flexural strength ranged from 817-1195 MPa for Y-TZP and 868 MPa for In-Ceram Zirconia. All specimens were polished with up to 4000-grit paper.[73] That is almost very difficult to do on a pre-sintered zirconia crown. Another study by Bhargava showed a high flexural strength value of 1039 MPa for Y-TZP when the specimens were air-abraded with alumina particles.[74]

The results in this study showed lower values than those in the above-mentioned studies. The non-translucent zirconia (CAP QZ) that was tested in our study showed 788

(44) MPa flexural strength, while the translucent zirconia showed lower values (558-714 MPa).

An explanation of the difference between our result and others might be due to differences in the surface treatment. Polishing protocols vary between this study (600-grit) and the above mentioned studies (1200- and 4000-grit). The processing condition in this study simulated the condition of a sintered crown after adjustment. Clinically, it is almost impossible to polish sintered zirconia crowns with 1200-grit or 4000-grit. On the other hand, sand-blasting the surface of zirconia bars causes transformation from the tetragonal to the monoclonal phase, thus producing compressive outer layers that cause crack propagation, leading to an increase in the strength of zirconia. This finding was investigated and confirmed by Kosmak et al.[75] and Guazzato et al.[76]

Fracture toughness is an intrinsic property describing the energy required to fracture the material when there is a flaw. Indentation, single-edge notch beam (SENB) and single-edge V notch beam (SEVNB) are common tests to measure the fracture toughness of dental ceramic. SEVNB is one of the most reliable, accurate, and reproducible methods to measure the fracture toughness of dental ceramics which also recommended by ISO 6872.[77]

Gogotsi et al. reported fracture toughness of 5.7 MP.m$^{1/2}$ when testing Y-TZP in SENVB method.[78] A similar value was obtained by Kubler and others (5.34 MPa.m$^{1/2}$).[79] Another study was run by Triwatana et al.[80] when comparing SEVNB fracture toughness of Y-TZP and In-Ceram Zirconia in a four point bending, 5.4 MPa.m$^{1/2}$ and 4.1 MPa.m$^{1/2}$ respectively.

In the present study, Suntech shows a comparable value to other studies of 5.42 $MPa.m^{1/2}$. The fracture toughness values for translucent and non-translucent were higher (6.08-6.86 $MPa.m^{1/2}$) than in previous studies.

The QZ (non-translucent zirconia) showed significantly higher flexural strength than translucent zirconia, and all zirconia (translucent and non-translucent) brands have significantly higher flexural strength and fracture toughness than e.max, as expected. So the first null hypothesis was rejected.

From the results of this study, it can be inferred that modification to enhance the translucency of non-translucent zirconia led to a decrease in the flexural strength. Another support for the result is that QZ (the non-translucent zirconia) and FZ (one of the translucent zirconia) are made by the same manufacturer, Custom Automatic Prosthetic. The manufacturer states in its website that the QZ has better mechanical properties than FZ.

IPS e.max crowns have gained popularity among dentists as they have shown excellent esthetic results and good clinical outcomes. But, 2-mm occlusal and 1.5-mm axial tooth reduction is required to have good restoration integrity. In some cases, this excessive reduction compromises the integrity of the prepared tooth. That is why full gold crowns are still being used because they require less reduction.

Translucent full-contour zirconia was introduced to offer a stronger restoration, requiring less tooth reduction while maintaining good optical quality. In order to study the clinical outcome of ceramic materials, crown-shape specimens are fabricated and tested in situations similar to the clinical environment. Since the materials that were used in this study are newly introduced, there is little information about their strength. In this study, zirconia crowns were fabricated with 1-mm overall reduction, while IPS e.max crowns were made with 2-mm and 1.5-mm occlusal and axial reduction. Epoxy resin was selected as die material because of its similar modulus of elasticity to dentin.[62] The crowns were loaded with a 4-mm rounded-end stainless steel at a 10-degree angle with a 1-mm tin pad in between to prevent the surface damage. All the crowns were fractured in the range of 2131 N to 2489 N. There was no significant difference between all groups regarding fracture resistance except for QZ and FZ, which were significantly higher than ST. That means 1-mm zirconia crowns have comparable fracture resistance to 2-mm e.max crowns. So the first part of null hypothesis was rejected, and the second part was partially rejected.

SUMMARY AND CONCLUSION

In the present study, the processing technique to increase zirconia translucency was found to cause a significant decrease in the fracture toughness and flexural strength, but not in the fracture resistance.

It was also observed that translucent zirconia has better mechanical properties than lithium disilicate.

Zirconia crowns with the recommended thickness showed strength comparable to that of lithium disilicate crowns with the recommended thickness.

The results of the present study indicate that tooth structure can be preserved by fabricating zirconia crowns instead of lithium disilicate crowns.

This study had limitations in its ability to simulate oral environmental changes. The loading was static instead of cyclic fatigue, and the moisture and the temperature of the oral cavity were not simulated. Also specimens were not thermal-cycled. Future studies are indicated that should simulate the oral environment, measure the fatigue load, and compare translucencies.

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ABSTRACT

MECHANICAL PROPERTIES OF

FULL-CONTOUR ZIRCONIA

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Background: Today, zirconia is used widely as core material for all-ceramic crowns. The core is usually veneered with a more translucent ceramic to provide a more esthetic restoration. Lately, several manufacturers claim that new translucent zirconia materials can be used as full-contour crowns without veneering, which would require less tooth reduction than is needed for lithium disilicate full-ceramic crowns. However, studies have not been done to verify this claim.

The objectives:

1. To compare the flexural strength, flexural modulus, and fracture toughness of specimens fabricated from recently marketed translucent full-contour zirconia, traditional zirconia, and lithium disilicate glass ceramic.

2. To compare the load-to-failure of crowns fabricated from recently marketed translucent full-contour zirconia, traditional zirconia, and lithium disilicate glass ceramic at their recommended tooth-reduction thickness.

Methodology: Four groups of translucent zirconia (BruxZir, KDZ Bruxer, CAP FZ, Suntech zirconia), one group of traditional zirconia (CAP QZ) and IPS e.maxCAD) were tested. Twelve bars of each material were made and tested for flexural strength, and fracture toughness. Fracture patterns were imaged under SEM. Forty-eight crowns (8 from each group) were fabricated with CAD/CAM technique following manufacturers' recommendations for the amount of tooth reduction. All the crowns were cemented to prepared epoxy resin dies with RelyX Unicem and tested for static load to failure in a universal machine.

Result: In bar-shape samples, CAP QZ (traditional zirconia) showed the highest flexural strength (788.12 MPa), fracture toughness (6.85 MPa.m1/2), and fracture resistance (2489.8 N). All translucent zirconia groups show lower mechanical properties than QZ. However, there were no differences between translucent and traditional zirconia in the fracture resistance of the crown-shape samples. There was no significant difference in fracture resistance between IPS e.max crowns at recommended thickness and other zirconia crowns at recommended thickness.

Conclusion: With less reduction of tooth structure, a high inherent strength and chip resistance make full-zirconia crowns a good alternative to porcelain-fused-to-metal crowns and all other ceramic crowns.

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