Influence of core thickness and artificial aging on the biaxial flexural strength of different all-ceramic materials: An in-vitro study

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The purpose of this study was to investigate the flexural strength of all-ceramics with varying core thicknesses submitted to aging. In-Ceram Alumina (IC), IPS e.max Press (EM) and Katana (K) (n=40), were selected. Each group contained two core groups based on the core thickness as follows: IC/0.5, IC/0.8, EM/0.5, EM/0.8, K/0.5 and K/0.8 mm in thickness (n=20 each). Ten specimens from each group were subjected to aging and all specimens were tested for strength in a testing machine either with or without being subjected aging. The mean strength of the K were higher (873.05 MPa) than that of the IC (548.28 MPa) and EM (374.32 MPa) regardless of core thickness. Strength values increased with increasing core thickness for all IC, EM and K regardless of aging. Results of this study concluded that strength was not significantly affected by aging. Different core thicknesses affected strength of the all-ceramic materials tested (p<0.05).

Keywords: All-ceramic, Flexural strength, Core thickness, Aging

INTRODUCTION

All-ceramics have been increasingly used in prosthetic dentistry to fabricate a wide variety of restorations1). The most important disadvantage of all-ceramic restorations is probably brittleness. This property is responsible for the strength behavior of all-ceramics. The limited capacity to undergo plastic deformation results in failure at the first sign of overloading2,3). Few of these materials hold adequate mechanical properties for clinical use as aesthetic all-ceramic single crowns and fixed prosthesis, when subjected to high stresses4,5). The most commonly used systems can be classified according to laboratory procedure used to obtain the core or fabrication (pressable, slip-casting, milling or sintering)6,7). The lithium disilicate reinforced glass ceramic, IPS e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein) is preferred due to the strength of the improved heat-pressed all-ceramic material, its superior biocompatibility and its good aesthetic features. However, mechanical properties of high performance alumina and zirconia based ceramics make them important as potential materials for all-ceramic restorations in high stress-bearing areas8). In-Ceram Alumina (Vita Zahnfabrik, Bad Sackingen, Germany), glass-infiltrated ceramic core employs the slip-casting technique, has been used as a core material for crowns and short-span bridges since the early 1990s9). The ceramic substructure of this system is extremely porous and composed of aluminum oxide, which is then infiltrated with fused glass. The high content of alumina particles combined with the low sinterization shrinkage, enhance the mechanical properties of the material10). The most recent core materials are the yttrium oxide partially stabilized zirconia (Y-TZP), were introduced as an alternative material for all-ceramic dental restorations that are manufactured into blanks and milled to the desired dimensions using the CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) technology11,12). Their transformation toughening characteristic leads to the increased fracture strength of Y-TZP ceramics compared with other all-ceramics13).

Clinically, an appropriate strength characteristics is an important aspect of dental restoration. According to previous studies, the long-term strength properties of all-ceramic restorations depend upon several factors, such as fabrication type14-17), laboratory procedures18-21), core or veneer thickness4,7,22), fatigue resistance23), luting procedure24,25), and aging11,14,16-28). Thus, it is important to determine the mechanical behavior of this category of dental materials in vitro.

Ceramic core-veneered fixed prosthesis have been used for several applications (anterior and posterior restorations), and these combine the strength of ceramic cores with the esthetics of the veneering porcelains29). Although the mechanical characteristics of core materials have continuously been improved, it is uncertain whether thickness change of the core material would necessarily result in the different flexural strength values of core/veneering ceramic systems.

Standart test methods for determining the flexural strength of ceramic materials are either uniaxial or biaxial flexural tests such as piston-on-ring, piston-on-three-ball, ball-on-ring and ring-on-ring tests30). Biaxial flexural strength tests have several advantages to
uniaxial tests. The measurement of strength of brittle materials under biaxial flexural conditions is often considered more reliable, because the maximum tensile stresses occur within the central loading area, and spurious edge failure are eliminated31-33).

Some studies showed a number of factors affecting the mechanical properties of the layered all-ceramic materials, such as temperature and moisture11,26,28,34). In the oral environment, all-ceramic materials are prone to aging which can lead all-ceramic materials to change color, to lower bending strength, and to reduce anti-fracture toughness. Artificial aging process simulates the effects of long-term exposure to environmental conditions through an artificial weathering process that involves light exposure, temperature and humidity35). Thus, it is possible that the mechanical characteristics of the all-ceramics are altered when subsequently loading them in an aqueous environment.

Although the strength of the all-ceramics is well documented, data on the effect of both varying core thicknesses and artificial aging on biaxial flexural strength of different all-ceramics is limited4,11,36). Therefore, the purpose of this study is to investigate in vitro the biaxial flexural strength of double-layer all-ceramic systems with different core thicknesses which have been subjected to an artificial aging process.

**MATERIALS AND METHODS**

Three types of core-veneered ceramics were fabricated, including the In-Ceram Alumina glass-infiltrated porcelain (IC) with VM7 as the veneer (Vita Zahnfabrik), the IPS e.max Press (EM) pressable ceramic with IPS e.max Ceram as the veneer (Ivoclar-Vivadent), and the zirconia core Katana (K) with CAD/CAM with CZR as the veneer (Noritake Dental, Nagoya, Japan). Forty disc-shaped, porcelain/ceramic bilayered specimens for each all-ceramic systems with a 10 mm diameter were prepared according to ISO specification 687237). Each all-ceramic group contained two core groups based on the core material thickness as follows: IC/0.5, IC/0.8, EM/0.5, EM/0.8, K/0.5 and K/0.8 mm in thickness (n=20 each). One point zero millimeter of veneer porcelain was applied on all the ceramic specimens. The materials and the groups are shown in Table 1. For each type of the core groups, ten specimens were randomly selected and subjected to artificial aging. All specimens were tested for flexural strength for each group (control and aged specimens).

**Preparation of the specimens**

An aluminum mold was fabricated which consisted of holes with different depths for the preparation of In-Ceram Alumina (IC) discs and duplicated with the investment (Vita In-Ceram Spezialgips, Bad Sackingen, Germany) with the help of vinyl polysiloxane impression material (Express XT, 3M, St.Paul, MN, USA). Discs were prepared according to the manufacturer’s instructions and removed from the mold with a diamond burr. Finishing procedures were performed and the final thicknesses of the core discs were verified as 0.5 and 0.8 mm (±30 μm) with a digital caliper (Alpha-Tools Digital Caliper, CA, USA). The alumina core discs were cleaned in an ultrasonic cleaner with distilled water. To prepare the standard 1.0 mm thick veneer porcelain, each disc was placed in a silicon mold (10-mm diameter, 1.5 and 1.8-mm depth). Veneering porcelain (Vita VM 7, Vita Zahnfabrik) was applied on the disc surface with a brush and fired according to the manufacturer’s instructions. Artifical aging process (Vita VM 7, Vita Zahnfabrik) was applied on the disc surface with a brush and fired according to the manufacturer’s instructions. The final dimensions for each disc were measured with a digital caliper.

Wax pattern discs for the IPS e.max Press (EM) specimens were fabricated using a stainless steel mold and invested in a phosphate-bonded investment cylinder, according to the manufacturer’s instructions. Investment cylinders were heated in a furnace for one hour at 850°C. IPS e.max Press medium opacity ingots (MO1) were selected for fabricating the ceramic discs. The specimens were heat-pressed (Ivoclar EP 600 Combi, Ivoclar Vivadent) at 920°C for 25 min. After the investment had cooled, the specimens were divested with airborne particle abrasion using 50 μm glass beads. The residual investment material on the ceramic discs was cleaned by dipping the discs in invex liquid (Invex Liquid, IvoclarVivadent), which contains less than 1% hydrofluoric acid, for 10 min. The specimens were then left in distilled water for 30 min. The thickness of each specimen was controlled with a digital caliper.

| Core Type                | Core thickness (mm) | Code | Brand name       | Manufacturer                   |
|-------------------------|--------------------|------|------------------|--------------------------------|
| Glass-infiltrated        |                    |      |                  |                                |
| aluminum oxide core      | 0.5                | IC   | In-Ceram Alumina | Vita Zahnfabrik, Bad Sackingen, Germany |
|                         | 0.8                |      |                  |                                |
| Lithium-disilicate       |                    |      |                  |                                |
| reinforced glass         | 0.5                | EM   | IPS e.max Press  | Ivoclar Vivadent, Schaan, Liechtenstein |
| ceramic core             | 0.8                |      |                  |                                |
| Yttrium stabilized       |                    |      |                  |                                |
| zirconia core            | 0.5                | K    | Katana           | Noritake Dental, Nagoya, Japan  |
|                         | 0.8                |      |                  |                                |
and reduced until the desired ceramic core thickness was achieved (0.5 or 0.8 mm). The veneering process was completed following the IC method: Veneering porcelain (IPS e.max Ceram, Ivoclar) was applied to the discs and fired in accordance with the manufacturer’s recommendations.

Katana (K) zirconium oxide discs with thicknesses of 0.5 or 0.8 mm were fabricated by milling pre-sintered KT13 zirconium blocks (94.4% ZrO₂, 5.4% Y₂O₃) according to the manufacturer’s instructions with the Dental Wings CAD/CAM system (DWOS, Montreal, Canada). The zirconium blocks were machined with 1.3 mm in diameter diamond burs in the CAM unit (Yenamak D50, Yenadent, Istanbul, Turkey). All machined discs were designed 21% larger than the desired size to compensate for sintering shrinkage. A digital caliper was used to control the thicknesses. After the milling process, the disc-shaped specimens were sintered at 1,400°C for two hours. CZR (Noritake Dental) was applied to the core discs as the veneer porcelain. A custom-made silicon index was again used to prepare zirconium oxide disc specimens with standard veneer thicknesses.

**Aging process**

Ten specimens for each core group were subjected to artificial aging procedure consisting of exposure to ultraviolet light and water spray in the weathering machine (Xenotest 150 S+, Atlas Electronic Devices, IL, USA) for 200 h. The back panel temperature varied between 70°C (light) and 38°C (dark), and humidity varied between 50% (light) and 95% (dark). The testing cycle consisted of 40 min of light only, 20 min of light with front water spray, 60 min of light only, and 60 min of dark with back water spray. The dry bulb temperature was 38°C (dark) and 47°C (light), and the water temperature was 50°C.

**Biaxial flexural strength test**

The flexural strength of the all-ceramic disc specimens were determined with the piston on three-ball test according to ISO 6872[^15], performed in a universal testing machine (Instron, Norwood, MA, USA) (Fig. 1). Disc specimens were supported on three stainless steel spheres (3.2 mm diameter) equally spaced on a circle with a diameter of 10 mm. A 0.5 mm thick plastic sheet was placed between the disc specimen and 1.2 diameter center-loaded piston. The loading surface was the veneer porcelain in the specimens and the core surface of the specimens was in the bottom. The specimens were loaded in the testing machine with a 5 kN load cell, at a crosshead speed of 0.5 mm/min until failure. The recorded fracture load (N) was used in conjunction with the following equation to give the flexural strength (MPa):

\[
S = -0.2387P(X - Y)/d^2
\]

S: biaxial flexural strength (MPa); P: fracture load (N); d: disc specimen thickness (mm)

X: \((1+\nu)(B/C)^2+(1-\nu)^2/2(B/C)^2\)

Y: \((1+\nu)(1+\ln(A/C)^2)+(1-\nu)(A/C)^2\)

\(\nu\): Poisson’s ratio (ceramic=0.25, ISO 6872); A: radius of the support circle (mm); B: radius of the loaded area (mm); C: radius of the disc specimen (mm)

**Statistical analysis**

All statistical analyses and calculations were completed using the SPSS 15.0 (SPSS, Chicago, IL, USA) statistical program. Differences in biaxial flexural strength of all-ceramic discs based on brand, core thickness or aging were analyzed. The Kolmogorov-Smirnov test was used to evaluate the distribution of the data. The Kruskal-Wallis multiple comparison test was used to analyze the effects of aging on the biaxial flexural strength of all-ceramic systems. The Wilcoxon signed rank test was used to evaluate the effects of core thickness within the groups. To compare flexural strength between two groups, the Bonferroni correction Mann Whitney U test was performed. Statistical significance was determined given a \(p\)-value less than 0.05.

**RESULTS**

Table 2 presents means and standard deviations for the biaxial flexural strength values of the IC, EM and K groups before and after aging. According to the results, the type of the ceramic influenced the flexural strength values. Statistically significant differences were observed between the mean strength values of the IC (548.28 MPa), EM (374.32 MPa) and K (873.05 MPa) groups using the Kruskal-Wallis multiple comparison test \(p<0.05\). Nonetheless, K group showed greater flexural strength than the IC and EM groups, EM had the lowest mean flexural strength values regardless of
Table 2  The strength parameters obtained from the biaxial flexural strength test with and without aging

| Property                  | Without aging | With aging |
|---------------------------|---------------|------------|
|                           | EM            | IC         | K           | EM            | IC         | K           |
| Range of strength (MPa)   | 225.67–556.15 | 589.12–660.55 | 512.14–1292.56 | 209.36–473.84 | 325.02–648.26 | 461.78–1395.89 |
| Mean (SD) strength (MPa)  | 374.32 (143.84) | 548.28 (90.99) | 873.05 (354.57) | 333.20 (114.64) | 528.17 (93.57) | 819.92 (352.05) |

Table 3  The mean biaxial flexural strength values (MPa) of all-ceramics compared with different core thickness with and without aging

| Material | Core thickness (mm) | Biaxial flexural strength |
|----------|---------------------|---------------------------|
|          |                     | Without aging             | With aging               |
|          |                     | Mean (SD)                 | Mean (SD)                |
| EM       | 0.5                 | 235.88 (11.15)a           | 224.24 (24.75)a          |
|          | 0.8                 | 512.75 (31.17)b           | 442.17 (27.36)b          |
| IC       | 0.5                 | 460.71 (8.56)b            | 442.82 (42.47)b          |
|          | 0.8                 | 635.86 (19.13)c           | 613.53 (22.18)c          |
| K        | 0.5                 | 531.04 (13.39)b           | 484.44 (17.54)b          |
|          | 0.8                 | 1215.06 (72.76)d          | 1155.41 (106.0)d         |

*aWithin any column and line means with the same letters are not significantly different (p>0.05). Different letters indicate significantly difference of groups (p<0.05).

Comparing the artificial aging procedure; it was shown that, slightly decreased flexural strength values of IC, EM and K after aging. Consequently, Wilcoxon test indicated that this difference in biaxial flexural strength values between aged and non-aged specimens was not significant regardless of core thicknesses (p>0.05).

The mean strength values (without aging, with aging) obtained for the core thickness groups were: EM/0.5 (235.88, 224.24 MPa), EM/0.8 (512.75, 442.17 MPa), IC/0.5 (460.71, 442.82 MPa), IC/0.8 (635.86, 613.53 MPa), K/0.5 (531.04, 484.44 MPa), K/0.8 (1215.06, 1155.41 MPa). The effect of core thickness on the strength values was statistically significant (p<0.05). For all groups, both 0.8 mm core groups had significantly higher mean biaxial flexural strength values than 0.5 mm core groups with and without aging. EM/0.8, IC/0.5, and K/0.5 core groups have shown similar strength values both with and without aging (Table 3).

Visual inspection of the specimens after flexural test presented that the cracks are constantly asymmetric and the main fracture type was splitting of the specimen. Observation of the fracture surfaces showed no signs of major interfacial delamination between the core and veneer material. Some specimens showed minor delamination, about 1 to 2 mm from the crack margin, while the crack origin was located at the center of the specimen (Fig. 2). In both systems, delamination and splitting occurred at the same fracture load. 0.8 mm core groups fractured in four or more pieces (4–5) while the other groups fractured in three or less (3–2). Especially the K group specimens fractured hardly and catastrophically under the applied stress. For EM

Fig. 2 Fracture of the specimen at the biaxial flexural testing machine.
and IC groups, there were no noticeable differences in the fracture type of the specimens compared to those obtained in the fracture surfaces.

**DISCUSSION**

Strength is an important mechanical characteristic that can assist in predicting the performance of brittle materials\(^4\). In the current study, the biaxial flexural strength of various thickness of all-ceramic core, and, conversely same thickness of the veneer ceramic were evaluated *in-vitro*. Specimens, which would have been fabricated according to the manufacturer's directions, and testing conditions were chosen carefully to imitate clinical condition as much as possible. Therefore an understanding of actual clinical strength behaviour of all-ceramics is absolutely necessary before results of *in vitro* strength testing can be considered to have clinical validity\(^9\). Considering the current results of flexural strength, the fracture load of clinical crowns for various all-ceramic materials, and the maximum occlusal load of 600 N in the clinical situation, it appears to be possible to lead appropriate choices when faced with various clinical conditions, even though the strength of crown depends on not only thickness, but also on shape, size, test method, load direction, adhesive cements.

According to the results of current study; the heat-pressed technique for lithium disilicate reinforced glass ceramic, the slip-casting technique and the CAD/CAM technique showed significantly different mean biaxial flexural strength values regardless of the core thickness and aging \((p<0.05)\). For this reason, the fabrication technique may have an effect on the mechanical properties of the tested materials. Current results are in agreement with the findings of several studies, in which reported that CAD/CAM fabricated zirconia were shown to have better mechanical characteristics due to microstructural nature compared to the slip casting ceramics and lithium disilicate reinforced glass ceramics\(^4,31,38\). Other factors about all-ceramics, such as clinical use, prosthetic restoration design (laminate, single crown or the posterior bridge) and also laboratory processing techniques should also be considered and can be further investigated in future studies.

The effect of specimen thickness is one of the most important factors in the determination of biaxial flexural strength\(^7\). In recent studies it has been reported that there is a correlation between the thickness of the core/veneer ceramics and the flexural strength\(^7,22,24,39\). On the other hand, Thompson *et al.*\(^40\), in a fractographic study of clinically failed crowns, concluded that fracture initiation sites of dental ceramics are controlled primarily by the location and size of the critical flaw and not by specimen thickness. Therefore it was emphasized that when it is beyond a specific core thickness for each ceramic material, increase in thickness had little effect on overall flexural strength of the material\(^4\). Lithium disilicate cores should be fabricated at a minimum thickness of 0.8 mm, and glass infiltrated and yttrium stabilized zirconia cores at 0.5 mm, according to the manufacturers’ recommendations\(^41\). In the current study, specimen core thicknesses were determined to be in 0.5 or 0.8 mm, and all the veneer thicknesses were 1.0 mm. Based on our findings, these variations in thickness would have significantly influenced the biaxial flexural strength values of the selected materials \((p<0.05)\). Strength values increased with increasing core thickness for all IC, EM and K groups. The results also implied that EM/0.8, IC/0.5, and K/0.5 core groups have shown similar strength values both with and without aging (Table 3). In the present study, a same veneer thickness (1.0 mm) was fabricated for all specimens to minimize the influence of the veneering porcelain on the measured strength. Although veneering parameters were eliminated the study, veneering porcelain was used onto the disc specimens, to imitate the clinical crown conditions.

Based on the previous color study; Dikicier *et al.*\(^42\) reported that all-ceramics color change was less influenced by increasing core thickness. The present study indicated that strength values increased with increasing core thickness. Consequently, comparing the color and strength properties of all-ceramics, core thickness is an important factor to determine where the all-ceramic systems is used clinically. The results may guide for prosthetic restoration design, when aesthetic is important, when occlusal forces are excessive or not.

Dental restorative materials must withstand widely varied conditions in the mouth, including temperature changes, continuous exposure to moisture, and mechanical use of the restoration. Light exposure and humidity changes can be simulated artificial aging process which has been widely used for the testing of dental resin and ceramic materials\(^11,43,44\). The manufacturer of the universal test machine used in this study claimed that 300 h of artificial aging is equivalent to 1 year of clinical service. Beuer *et al.*\(^36\) studied the strength of CAD/CAM fabricated all-ceramics with similar aging procedure. They reported that no difference was found in the strength values with or without aging. On the other hand, Flinn *et al.*\(^46\) revealed that aging for 200 h can cause significant transformation from tetragonal to monoclinic crystal structure, which results in a statistically significant decrease especially in the flexural strength of Y-TZP. In the current study, all-ceramic systems were artificially aged for 200 h to evaluate strength changes. The aging process resulted in a slight decrease in biaxial flexural strength of IC, EM and K groups (Table 3). Nevertheless, strength values of the all specimens not influenced by aging \((p>0.05)\). This results is consistent with a similar *in-vitro* study, investigating the fracture strength of Y-TZP ceramics with aging\(^27\). Although the aging time likely to be experienced *in vivo* has not been determined, a provisional estimate of oral conditions was suggested. Two hundred hours of the present study might simulate relatively short period, therefore longer lifetime effect of the all-ceramics should be further studied.

Although the method of specimen fabrication is important, the strength test method employed will
affect the results\textsuperscript{30}. The present study used the biaxial flexural test with piston-on-three-ball method which has the reliable technique for studying brittle materials and standardized by ISO\textsuperscript{7,31,32,37,40}. In this in-vitro study disc-shaped specimens of two different core thicknesses were fabricated out of all-ceramic materials. Biaxial flexural strength was compared using an artificial aging process to simulate oral environmental conditions. It is important to emphasize that the aging process used in this study is only a first step toward predicting clinical performance. Further in vivo studies should be performed on the clinical evaluation of core thickness and flexural strength for better characterization of all-ceramics.

CONCLUSIONS

Within the limitations of the present study; following conclusions were drawn:

1. According to the biaxial flexural test, the K group had significantly stronger than the other all-ceramic groups, and the EM group had lower strength values regardless of core thickness and aging.

2. Aging did not have a significant effect on the biaxial flexural strength of the selected all-ceramics.

3. Biaxial flexural strength of all-ceramics were affected by core thicknesses. In addition, the double-layered specimens with different ceramic core thickness showed a decrease in strength could be attributed to the reduction of core thickness.

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