Effect of mechanical cycling on the flexural strength of densely sintered ceramics

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ABSTRACT

Objectives. The aim of this study was to evaluate the effect of mechanical cycling on the biaxial flexural strength of two densely sintered ceramic materials.

Methods. Disc shaped zirconia (In-Ceram Zirconia) and high alumina (Procera AllCeram) ceramic specimens (diameter: 15 mm and thickness: 1.2 mm) were fabricated according to the manufacturers’ instructions. The specimens from each ceramic material (N=40, n=10/per group) were tested for flexural strength either with or without being subjected to mechanical cycling (20,000 cycles under 50 N load, immersion in distilled water at 37 °C) in a universal testing machine (1 mm/min). Data were statistically analyzed using two-way ANOVA and Tukey’s test (α=0.05).

Results. High alumina ceramic specimens revealed significantly higher flexural strength values without and with mechanical cycling (647 ± 48 and 630 ± 43 MPa, respectively) than those of zirconia ceramic (497 ± 35 and 458 ± 53 MPa, respectively) (p<0.05). Mechanical cycling for 20,000 times under 50 N decreased the flexural strength values for both high alumina and zirconia ceramic but it was not statistically significant (p>0.05).

Significance. High alumina ceramic revealed significantly higher mean flexural strength values than that of zirconia ceramic tested in this study either with or without mechanical cycling conditions.

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1. Introduction

Dental restorative appliances should meet the requirements for mechanical strength, longevity and aesthetics in the oral environment. Historically, all-ceramic restorations fabricated with conventional feldspathic ceramics, had shorter longevity compared to porcelain-fused-to-metal fixed partial dentures (FPD), due to their low strength. In an attempt to overcome this problem, ceramic technology introduced infiltrated or densely sintered ceramic materials that allow for greater longevity of such restorations. In vitro studies revealed that densely sintered ceramics have the ability to resist against the compressive and shear forces [1,2]. The great advantage of these ceramics is the elimination of metallic frameworks resulting in natural appearance of the dental tissues.

One such system uses aluminum oxide framework that may be reinforced by a baking technique involving glass infiltration and making it possible to fabricate three-unit FPDs for anterior and posterior restorations with high flexural strength and excellent marginal adaptation comparable to their metallic counterparts [3,4]. The ceramic framework of this system is initially extremely porous and composed of aluminum...
oxide, which is then infiltrated with fused glass by capillary action that also allows for selection of the colour of the substructure. The high content of alumina particles with dimensions of 0.5–3.5 μm, combined with the low sinterization shrinkage, enhance the mechanical properties of the material [5]. With regard to the indication of three-unit FPDs at the posterior region, the In-Ceram Zirconia system was developed to withstand greater loads due to aluminium oxide and zirconia in its composition. Zirconia has tetragonal crystals that allow application of external mechanical energy on the material. The energy is then, transformed in the monoclinic form of zirconia, thus avoiding propagation of microcracks in the ceramic and providing good sealing of the material [6,7].

Other densely sintered ceramic systems that are also designed without metallic framework are often used in combination with CAD-CAM technology (Computer Assisted Design–Computer Assisted Machining/Manufacturing), supplying a framework made of sintered, densely compacted 99.5% pure aluminium oxide. Sintered pure aluminium oxide is characterized as a bioceramic presenting biaxial flexural strengths close to 687 MPa, yielding relevant mechanical properties for application in single unit restorations or FPDs at the anterior and posterior regions [8–9]. The new ceramic systems without metal substructure provide a relevant potential as an alternative to metal substructure. The high content of alumina particles with satisfactory mechanical properties compared to metal substructure [4] However, some factors such as high forces and repetitive stresses during the chewing cycle may lead to fatigue of the material and eventually fractures when they are exposed to the oral environment [10]. Fatigue tests [10,11] are very important in order to evaluate the mechanical performance of ceramic materials and to provide more information than studies addressing a constant loading rate [12–15].

The forces applied on the materials in the oral cavity develop cyclic loads that can be simulated by mechanical cycling that tends to be close to the physiological conditions generated by the chewing cycle [16–24]. Therefore, the objective of this study was to verify the effect of mechanical cycling for induction of fatigue and then to quantify the biaxial flexural strengths of zirconia and alumina reinforced ceramic framework materials.

2. Materials and methods

Two ceramic materials indicated for fabrication of FPD frameworks, namely the In-Ceram Zirconia system (Vita Zahnfabrik, Bad Sackingen, Germany) and the Procera AllCeram system (Nobel Biocare/Procera Sandvik AB, Stockholm, Sweden) were used for the experiments.

Ten disc shaped metallic molds with 15 mm inner diameter and 1.4 mm thickness, machined from an aluminium rod were used for the fabrication of ceramic specimens.

2.1. Specimen preparation for In-Ceram Zirconia

Metallic molds were fixed on a glass slab surrounded by wax support and the refractory investment, In-Ceram Spinell (Vita Zahnfabrik), was poured on the molds. The disc specimens created in the mold were coated with the lubricant Sealer Vita In-Ceram Zirconia (Vita Zahnfabrik). Thereafter, the spaces were filled with the zirconia oxide material of the In-Ceram Zirconia system in the aqueous phase, called slip casting. Sintering was performed in an oven (Inceramat, Vita Zahnfabrik) according to the cycle recommended by the manufacturer.

After completion of the cycle and cooling, the specimens were removed from the investment by grinding the mold with diamond burs at low-speed, without water cooling. The sintered zirconia surfaces were porous structures with chalk-like consistency. For that reason, the surfaces were ground finished to 1000 grit silicone carbide abrasive until 1.3 mm thickness was achieved. A digital micrometer (Absolute system, Mitutoyo, Suzano, SP, Brazil) was used to control the thickness of each specimen. In this phase, the zirconia oxide structures have low resistance and thus should be carefully handled until glass infiltration which was performed in the aforementioned oven for sintering. Glass infiltration comprises application of a layer of mixture of glass powder of In-Ceram Zirconia and distilled water that is applied evenly with a brush. This procedure was performed on one side of the disc allowing air escape through the opposite side during infiltration process. During this step, the specimens were placed on a platinum slab.

The zirconia oxide specimens were then air particle abraded with 50-μm aluminium oxide particles under a pressure of 2 bar at a distance of approximately 2 cm in order to remove the excess glass. Glass infiltration was further performed on the opposite side in the same manner.

2.2. Specimen preparation for Procera AllCeram

The other material evaluated in this study was the Procera AllCeram system (Nobel Biocare/Procera Sandvik AB, Stockholm, Sweden) for which a CAD-CAM technology was employed for the fabrication of sintered aluminium oxide frameworks. The process of fabrication of this ceramic material comprises reading by a scanner connected to a computer which constructs mapping of the surface and provides the image in three dimensions. The images captured were sent via modem to Procera Sandvik AB in Stockholm, Sweden, where the materials were machined.

The aforementioned disc shaped metallic molds with 15 mm diameter and 1.4 mm thickness were used for the images scanned by the Procera Scanner (Nobel Biocare). The metallic mold was fixed in this reading device on a platform that was rotated around an axis on which a sapphire tip with 2-mm diameter performed reading by contact, registering the 360° of circumference of the metallic mold. After each rotation, the probe was automatically elevated in 200-μm by the computer and a new line was traced until the entire structure was mapped with nearly 50,000 points.

The reading points captured and observed in three-dimensional images were then transmitted to the Procera Sandvik AB for reproduction of the refractory cast with an approximate increase of 20% in the dimensions, due to the need to compensate for the sintering shrinkage of aluminium oxide that is nearly 15–20%. First, the aluminium oxide powder was packed on the cast, then the external contour was provided by a bur system before sintering at 1550–1650°C for 1 h.
All specimens obtained were further ground finished to 2000 grit silicon carbide abrasive and polished using 2.5 μm aluminium oxide polishing paste, yielding to uniform structures with final dimensions of 1.2 mm in thickness and 15 mm in diameter.

2.3. Mechanical cycling

Twenty specimens for each system were randomly divided into two subgroups and subjected to mechanical cycling prior to biaxial flexural strength test.

Mechanical cycling of the ceramic materials was performed in a mechanical cycling machine (custom made, Paulista State University, Dental School, UNESP, Sao Jose dos Campos, Brazil) that was developed to simulate the mechanical forces generated during the chewing cycle.

The specimens were placed in a metallic base having three balls with 3.2 mm diameter, equidistant to each other, forming a plane. An upper rod with a 1.6 mm diameter tip was fixed on the plier that induced 50 N loads for 20,000 times, with a frequency of 1 cycle per second (Fig. 1). The device for testing was placed on the machine base that contained a thermostat to allow testing in aqueous medium at a constant temperature of 37 °C.

Ten specimens per ceramic type were submitted to the flexural strength testing in a universal testing machine (Instron 4301, Instron Corp., Norwood, MA, USA) where the load was applied at a constant speed of 1 mm per minute until fracture occurred.

The formula according to the guidelines of ISO 6872 was followed for the calculation of the data obtained from the biaxial flexural strength test [25].

Data were then statistically analyzed using two-way ANOVA and Tukey’s test (α = 0.05).

2.4. Statistical analysis

Statistical analysis was performed using SAS System for Windows, release 8.02/2001 (Cary, NC, USA). Statistical assumptions were evaluated before statistical analysis conducting a normality test. The means of each group were analyzed by two-way analysis of variance (ANOVA), with biaxial flexural test as the dependent variable and the ceramic types and mechanical cycling as the independent factors. P values less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made by Tukey’s adjustment test.

3. Results

The results of normality tests indicated that the residual values were normally distributed when plotted against predicted values. The uniformity and normality tests did not violate the ANOVA assumptions (Fig. 2a and b). The statistical power of the performed test at α = 0.05 was 0.346 for cycling and 0.05 for ceramics versus cycling.
under 50 N was not statistically significant (p > 0.05) between the groups with and without cycling with means of 638 ± 43 and 647 ± 48 MPa for high alumina and 458 ± 53 and 497 ± 35 MPa for zirconia ceramic, respectively.

Considering only the ceramics tested in this study, significant differences were observed (p < 0.05) with means of 639 and 477 MPa for high alumina and zirconia ceramic, respectively (Fig. 3).

High alumina ceramic specimens revealed significantly higher flexural strength values without and with mechanical cycling (647 ± 48 and 630 ± 43 MPa, respectively) than those of zirconia ceramic (458 ± 53 and 497 ± 35, respectively) (p < 0.05). The decrease in flexural strength values for both high alumina and zirconia ceramic after mechanical cycling for 20,000 times under 50 N was not statistically significant (p > 0.05).

4. Discussion

The mechanical strength of dental materials has been addressed in several studies. This concern lies on the fact that the oral environment may alter them physiochemically. The presence of moist and thermal changes together with repetitive mechanical efforts that are generated during the chewing cycle provides conditions for occurrence of degradation and fatigue. This is even more critical when ceramic materials are employed for restoration, especially due to their friability and low flexural strength.

Ceramics are susceptible to fatigue, and the accumulation of microstructural damages during mastication may lead to catastrophic fracture [18,19]. Ceramic restorations are often investigated for durability using quantitative fractography tests that presented cracks on the internal surface of the occlusal region on which the greatest stress was applied during the chewing cycle [20].

The incidence of intermittent loads on the same site may induce fatigue in ceramic materials. Fairhurst et al. [15] and Wiskott et al. [20] stated that failure because of fatigue is explained by the appearance of microscopic cracks in areas of load concentration. With the repeated incidence of loads, these cracks fuse with the preexisting fissures, thereby weaken the bulk material. Therefore, fracture is the result of cycles of forces that exceed the mechanical strength of the material.

Mechanical cycling of ceramic materials performed in this study could be considered as a good simulation of clinical situations. In some previous studies mechanical cycling was performed arbitrarily. Drummond et al. [2] employed mechanical stimuli for 1000 cycles with an initial load of 4 kg. Sobrinho et al. [21] submitted three groups of ceramics to mechanical cycling for 1–10,000 cycles, with loads of 20–300 N at a frequency of 1 Hz. Kheradmandan et al. [24] on the other hand, observed that mechanical cycling with a load of 25 N and frequency of 1.5 Hz led to fracture of the ceramic material between 40,400 and 310,000 cycles. However, Ohyma et al. [22] employed a maximum load of 4.93 N at a frequency of 20 Hz for 100,000 cycles and they observed that fracture may occur between 1000 and 100,000 cycles and there was formation of cracks already between 100 and 10,000 cycles. Therefore, since the aim of this study was to evaluate the effect of mechanical cycling on ceramics without fracture of the specimens prior to flexural tests, it was decided to perform 20,000 cycles with 50 N load.

For the materials investigated (Procera AllCeram and In-Ceram Zirconia), the mechanical cycling factor yielded a reduction in the flexural strength from 489 to 442 MPa. The statistical power of the performed test at w = 0.05 was 0.346 for cycling and 0.05 for ceramics versus cycling. Myers et al. [19] and Drummond et al. [2] evaluated the flexural strength of different ceramic materials with and without cyclic loads until fracture in dry medium and in water. They concluded that the presence of water led to a reduction in the flexural strength.

Based on this finding, in this study, the tests were performed in water.

In the most recent ceramic systems used for frameworks of FPDs, the ceramic compositions include different mechanisms to minimize the dissipation of forces generated during chewing. Each system is composed of particles incorporated to the matrix, thus redirecting the energy in relation to its plane, and limiting the propagation of possible cracks. The dental ceramics evaluated in this study, present different microstructure

![Fig. 3 – The mean biaxial flexural strength values (MPa) for high alumina and zirconia ceramic with and without mechanical cycling.](image)
and performance than conventional feldspathic ceramics that are basically composed of glass. The failures most commonly found in the latter are related to the presence of the crystalline phase in the glassy matrix [16]. The high alumina ceramic tested (Procera AllCeram) is sintered at high temperature that comprises an extremely compact sintered aluminium oxide. Compaction of the crystals allows a high flexural strength close to 687 MPa, and absence of porosity [12]. The mean biaxial flexural strength values achieved in this study without mechanical cycling (647 MPa) were close or higher than those of the mean values achieved in the studies of Zeng et al. [9] (634.5 MPa), Wagner and Chu [12] (687 MPa), Wtn et al. [15] (672 MPa) and Esquivel-Upshaw et al. [14] (323.4 MPa), all of which used the same method for establishment of the strength. In one other study, Zeng et al. [9] employed the ring-over-ring method but still found a flexural strength of 669.4 MPa for the Procera AllCeram.

The In-Ceram Zirconia system has 35% of zirconia crystals that significantly enhance the mechanical properties of this ceramic [7]. Zirconia crystals present a tetragonal design and when submitted to stress, it undergoes transformation of its phase to monoclinic crystal, yielding particles 3-5% greater. The volumetric increase results in local compression stresses between the surface crystals providing higher strength against the propagation of cracks [7]. Sorensen et al. [9] reported flexural strength of In-Ceram Zirconia with 700 MPa that is higher than observed in the present study, namely 646 MPa for the group without mechanical cycling and 457 MPa with mechanical cycling. This discrepancy could be due to the differences of methodologies employed in each research. Thus, it is very important to be careful when authors make comparisons among results from different research. In this study, the method adapted was the one recommended by International Standard Organization [25] because the test allows standardize the specimens thickness, diameter, shape and roughness. The results achieved from both ceramics tested in this study, with or without mechanical cycling, met the requirements of ADA specifications that recommends a minimum flexural strength value of 100 MPa for this type of ceramic restorative materials. It is however still questionable whether this value of flexural strength is sufficient in clinical practice when such ceramics are employed.

Although early studies and clinical outcomes allow indications of these ceramics, clinical failures related to reinforced ceramics are still being reported. Investigations are warranted to allow their indication more safely since even if it was not statistically significant, a reduction in flexural strength was observed after accomplishment of 20,000 cycles of mechanical stimuli.

5. Conclusions

Based on the results of this study, the following conclusions can be drawn:

(1) Mechanical cycling for 20,000 times reduced the biaxial flexural strength of the high alumina and zirconia ceramics tested but the effect was not statistically significant.

(2) High alumina ceramic revealed significantly higher flexural strength values than those of zirconia ceramic either with or without mechanical cycling conditions.

References


