Assessing Flexural Strength Degradation of New Cubic Containing Zirconia Materials

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ABSTRACT

Aim: Newer zirconia materials may have greater strength degradation under cyclic fatigue with increased yttria and cubic content. The purpose of this study was to evaluate the flexural strength (FS) degradation of newer zirconia materials compared to more traditional tetragonal zirconia materials.

Materials and methods: The following materials were tested: two 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) materials (Lava Plus, 3M ESPE; Katana ML, Kuraray), one 4 mol% partially stabilized zirconia (4Y-PSZ) material (Katana STML, Kuraray), two 5 mol% partially stabilized zirconia (5Y-PSZ) materials (Katana STML, Kuraray; Lava Esthetic, 3M ESPE), and one lithium disilicate material (IPS e.max CAD LT, Ivoclar Vivadent). Thirty beams were milled for each ceramic material with final dimensions of $4.0 \times 1.3 \times 18.0$ mm after sintering or crystallization. Each specimen was placed on a 3-point bend test device on a universal testing machine (Instron, Norwood, MA). Flexural strength was determined on 10 beam specimens per group with a central load applied until fracture. Flexural fatigue strength was then measured on the remaining 20 beam specimens per group using the staircase method for 6,000 cycles at 2 Hz. Data were analyzed with one-way ANOVAs/Tukey *post hoc* tests ($\alpha = 0.05$).

Results: A significant difference was found between groups ($p < 0.001$) per property. The 3Y-TZP zirconia materials had the greatest flexural and flexural fatigue strength. The cubic containing zirconia materials performed more moderately. The lithium disilicate material had the lowest strength values. The percent degradation in flexural fatigue strength of the 3Y-TZP zirconia materials was less than the 5Y-PSZ, Katana UTML, and the 4Y-PSZ, Katana STML, cubic containing materials, but similar to the 5Y-PSZ cubic containing material, Lava Esthetic.

Conclusion: The amount of strength degradation was material dependent, with the 4Y-PSZ or 5Y-PSZ cubic containing zirconia materials demonstrating greater or similar strength degradation compared to the primarily tetragonal 3Y-TZP zirconia materials.

Clinical significance: The differences in FS degradation between cubic containing materials and traditional zirconia materials could significantly impact the long-term success of these newer materials. Clinicians should be aware that these cubic containing materials may perform differently long-term than the very strong traditional 3Y-TZP materials and to follow manufacturer instructions on required material thickness and indications for use to prevent premature failure of the restoration.

Keywords: Degradation, Flexural strength, Zirconia.

INTRODUCTION

Zirconia restorations have been widely used since their introduction in 2004 for fixed dental prosthetics applications.[1] With the increased utilization of all-ceramic crowns, zirconia has been a popular choice due to its superior strength and toughness when compared to all other dental ceramics, especially for multiunit fixed dental prostheses.[2] Although there is a lack of long-term clinical studies quantifying failure rates of monolithic zirconia, it is suggested that they fracture at a relatively low rate.[3] Zirconia is undoubtedly strong, however, the limited translucency of these restorations have historically been a drawback when selecting a restorative material for esthetic cases.[4] Thus, manufacturers have continually focused their efforts towards improving the esthetic properties of zirconia.

In its pure form, zirconia can exhibit three phases; monoclinic, tetragonal, and cubic. While the stability of these phases is dependent on increasing temperature, it is possible to achieve each phase at room temperature by adding stabilizing oxides. The most widely used form of zirconia for dental applications is in the tetragonal phase which is a high temperature phase stabilized by adding 3 mol% yttria and therefore known as 3% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP). In this phase, zirconia demonstrates transformation toughening which results in a high flexural strength (FS). Transformation toughening occurs when sufficient stress is placed onto an yttria-stabilized

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zirconia prosthesis. In the stressed area, the tetragonal phase zirconia crystals can transform to the monoclinic form which causes a 3% volume expansion at the site of stress and halts crack progression.[5] This allows for the material to exhibit a biaxial FS of 900–1,200 MPa.[6] To compare, the 3-point FS of lithium disilicate is around 250–400 MPa and as high as 500 MPa (biaxial FS) as recently reported by the manufacturer.[7,8]

Although tetragonal zirconia has proven to be a very strong material, its limited translucency results in a less esthetic finished product.[9] Manufacturers have improved the formulation of zirconia by lowering the alumina content and controlling the zirconia grain size and processing density to achieve an increase in translucency.[10] Coloring is often utilized to further improve esthetics through the addition of metal oxides or tinting of milled restorations but either of these coloring methods may result in a further reduction of translucency.[11] Although there has been some success in increasing the translucency and esthetics using these methods, they have yet to provide a result equal to that of glass ceramics.[12]

Currently, focus has been to improve translucency of zirconia through the utilization of a significant cubic crystalline phase interspersed with the tetragonal phase.[13] With the abundance of these newer "high-translucency" zirconia on the market, some confusion exists between the identification and utilization of cubic zirconia in these products. Products listed as "high-translucency" zirconia have been found to describe both 3Y-TZP as well as the cubic zirconia materials, which have been defined as 4 mol% and 5 mol% yttria partially-stabilized zirconia (4Y-PSZ and 5Y-PSZ). This critical differentiation is necessary as the newer zirconia products that utilize the cubic phase at a higher ratio do not exhibit the transformation toughening exhibited by the more readily available 3Y-TZP materials.[9] This results in a significantly lower biaxial FS ranging from 500–700 MPa, or roughly 30–40% less FS of that found in 3Y-TZP.[7] Despite the reduction in strength, many manufacturers advertise these cubic zirconia restorations to have both the translucency and strength to be used in single and multiunit restorations anywhere in the mouth.[7] Although this has yet to be proven, it appears the major advantage of utilizing the strength of zirconia in a multiunit fixed dental prosthesis may be sacrificed for greater translucency in the material.

The manufacturer of Lava Esthetic (3M ESPE, St. Paul, MN), a 5Y-PSZ material, reports the highest biaxial FS of the cubic zirconia materials available at 800 MPa.[14] Lava Esthetic is advertised to be utilized as crowns, 3-unit fixed dental prostheses, inlays, onlays, and veneers. Katana UTML, a 5Y-PSZ material (Kuraray Noritake Dental Inc.), with an advertised biaxial FS of 557 MPa, is presented to be most suitable for full-contour restorations in the anterior region, but can be considered for a posterior single unit crown.[15] Katana STML (Kuraray Noritake Dental Inc.), a 4Y-PSZ material, reports an average biaxial FS of 748 MPa and is advertised for fabrication of FDP frameworks, FDPs, crowns, inlays, onlays, and veneers.

With the loss of transformational toughening, the mechanical properties of cubic containing zirconia materials have changed substantially.[9,16] Fatigue behavior and strength degradation of the newer cubic containing zirconia materials may have a greater significance in clinician's decisions to use them in scenarios where traditionally, the strength of zirconia was not in question. This is particularly true for multiunit posterior prostheses.

Limited research has been published evaluating FS degradation of these newer cubic zirconia materials. The purpose of this study is to evaluate the FS and the flexural fatigue strength and degradation of two 5Y-PSZ materials, one 4Y-PSZ material, two 3Y-TZP materials, and one lithium disilicate glass ceramic material. The null hypothesis is that there will be no difference in properties based on ceramic material.

Materials and Methods

The study was conducted in the United States Air Force Dental Research and Consultation Service at Joint Base San Antonio Fort Sam Houston, Texas, USA. The following materials were tested: two 3Y-TZP materials (Lava Plus with A2 dyeing liquid, 3M ESPE; Katana ML Shade A Light, Kuraray Noritake Dental Inc.); one 4Y-PSZ (Katana STML Shade A2, Kuraray Noritake Dental Inc), two 5Y-PSZ (Lava Esthetic Shade A2, 3M ESPE; Katana UTML Shade A2, Kuraray Noritake Dental Inc.); and one lithium disilicate material (IPS e.max CAD LT, Shade A2, Ivoclar Vivadent).

Specimen Preparation

Thirty specimens were prepared for each ceramic material for a total of 180 specimens. A CAM (computer-aided manufacturing) machine (I-Mes iCore 450i, Eiterfeld, Germany) was used to mill the zirconia beams out of the zirconia blanks. The beams were designed using DS SolidWorks software (SolidWorks, Waltham, MA) and the file was imported into Sum 3D, iCAM V5 milling software (I-Mes, iCore). The final size of the beam specimens was 4.0 mm in width, 1.3 mm in depth, and 18.0 mm in length after sintering in a furnace (Programat S1 1600, Ivoclar Vivadent) according to the manufacturer's instructions. The IPS e.max CAD beam specimens were milled from blocks in a MCXL milling device (Dentsply/Sirona, Charlotte, NC) using the Omnicom software (Version 4.4.4; Dentsply/Sirona) and crystallized in the Programat P500 furnace (Ivoclar Vivadent) according to the manufacturer's instructions. The surface of each specimen was polished with 600 and 1,000 grit polishing paper.

Flexural Strength

Flexural strength testing was completed in accordance with the international standard for ceramic materials ISO (International Organization for Standardization) 6872:2015. Ten beam specimens per group were fractured in a universal testing machine (Model 5543, Instron, Norwood, MA). Each specimen was placed on a 3-point bending test device, which was constructed with a 15.0 mm span length between the supporting rods (Fig. 1). The central load was applied with a head diameter of 2.0 mm at a crosshead speed of 1.0 mm/minute. The FS was obtained using the equation $FS = 3Fl/2bd^2$, where F is the loading force at the fracture point, l is the length of the support span (15 mm), and b is the width and d the depth of the beam specimen. Measurements were made using electronic digital calipers (GA182, Grobet Vigor, Carlstadt, NJ).
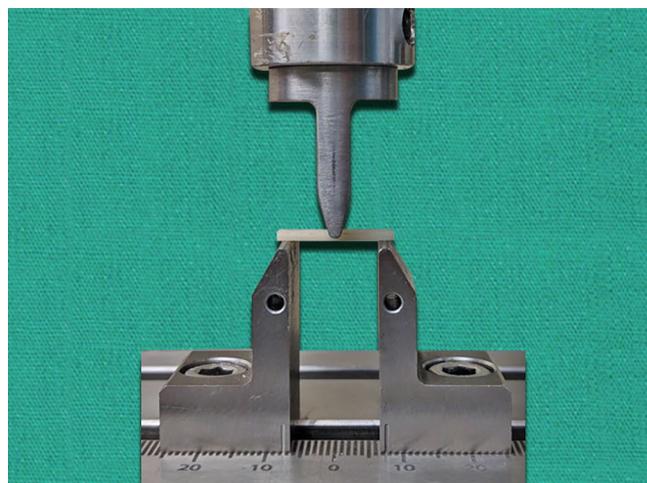

Fig. 1: Specimens placed on a 3-point bending device for flexural strength and flexural fatigue strength testing

Flexural Fatigue Strength

The flexural fatigue strength (σ_{ff}) of twenty zirconia beams was determined for 6,000 cycles at 2 Hz per group. Each flexural fatigue strength specimen was placed on the 3-point bending test device as was done with FS testing (Fig. 1). The staircase method was used for the fatigue resistance evaluation.[17] For each group, the starting force value was determined by using 1/2 of the maximum FS. The amplitude (stress alternating) was determined by using 1/2 of the standard deviation of the maximum FS. Tests were conducted sequentially with force applied values increasing or decreasing by 20% of initial starting force value whether the previous beam resulted in failure or survival. The flexural fatigue strength and standard deviation were determined using the following equations:

$$\sigma_{ff} = X_0 + d\left(\frac{\sum in_i}{\sum n_i} \pm 0.5\right)$$

$$SD = 1.62d\left(\frac{\sum n_i \sum i^2 n_i - \left(\sum in_i\right)^2}{\left(\sum n_i\right)^2} + 0.029\right)$$

X_0 is the lowest stress level considered in the analysis and d is the fixed stress increment. To determine σ_{ff}, the analysis of the data were based on the least frequent event (failures vs survivals). The negative sign is used when the analysis is based on failures; otherwise the positive sign is used. In the second equation, the lowest stress level considered is designated $i = 0$, the next $i = 1$, and so on, and n_i is the number of failures or survivals at the given stress level.[17]

Statistical Analysis

The mean and standard deviation for FS and flexural fatigue strength was calculated for each of the ceramic materials. Percent FS degradation was calculated based on the loss in FS after fatigue loading. Data were analyzed using one-way ANOVAs and Tukey *post hoc* tests for FS and flexural fatigue strength ($\alpha = 0.05$).

Results

3-point Bend Flexural Strength

The highest FS was found in the 3Y-TZP Lava Plus (870.6 ± 145.8 MPa) which was not significantly different from the 3Y-TZP Katana ML (777.9 ± 101.1 MPa). Both 3Y-TZP materials were significantly greater than the 4Y-PSZ Katana STML (534.3 ± 63.6 MPa), the 5Y-PSZ Lava Esthetic (485.0 ± 63.1 MPa) and the 5Y-PSZ Katana UTML (470.2 ± 42.9 MPa), which were not significantly different from each other. The lithium disilicate IPS e.max CAD had the lowest FS (262.9 ± 27.1 MPa) and was significantly lower than all the other materials (Table 1).

3-point Bend Flexural Fatigue Strength

The highest flexural fatigue strength was found with the 3Y-TZP Lava Plus (640.2 ± 129.8 MPa) which was significantly higher than the 3Y-TZP Katana ML (536.6 ± 158.8 MPa). The 4Y-PSZ and 5Y-PSZ materials performed more moderately and were all significantly less than the 3Y-TZP materials. The 5Y-PSZ Lava Esthetic (336.2 ± 48.2 MPa) was significantly greater than the 5Y-PSZ Katana UTML (232.2 ± 19.4 MPa). However, both of these groups were not significantly different from 4Y-PSZ Katana STML (304.4 ± 37.3 MPa). The lithium disilicate IPS e.max CAD had the lowest flexural fatigue strength

Table 1: Mechanical properties of tested materials. Groups with the same lower-case letter per column are not significantly different ($p > 0.05$)

Material	Content	Flexural strength (MPa)	Flexural fatigue strength (MPa)	Strength degradation (%)
IPS e.max CAD	Li₂Si₂O₅	262.9 (27.1)[a]	146.4 (24.0)[a]	44.3
Katana UTML	5Y-PSZ	470.2 (42.9)[b]	232.2 (19.4)[b]	50.6
Lava Esthetic	5Y-PSZ	485.0 (63.1)[b]	336.2 (48.1)[c]	30.7
Katana STML	4Y-PSZ	534.3 (63.6)[b]	304.4 (37.3)[bc]	43.0
Katana ML	3Y-TZP	777.9 (101.1)[c]	536.6 (158.8)[d]	31.0
Lava Plus	3Y-TZP	870.6 (145.8)[c]	640.2 (129.8)[e]	26.5

(146.4 ± 24.0 MPa) and was significantly lower than all the other materials (Table 1).

The percent degradation in flexural fatigue strength of the 3Y-TZP materials, Lava Plus (26.5%) and Katana ML (31.0%) was less than the 5Y-PSZ Katana UTML (50.6%) and the 4Y-PSZ Katana STML (43.0%), but similar to the 5Y-PSZ Lava Esthetic (30.7%) (Table 1).

Discussion

The null hypothesis was rejected because the results of this study demonstrate that there are significant differences in properties based on ceramic materials. The data shows similar trends between FS as those from previous studies by Zhang et al. and Kwon et al.[9,18] In this study, the 3-point bend FS data of the 4Y-PSZ and 5Y-PSZ groups (534 MPa, 470 MPa, 485 MPa) was 38–46% less than that of the 3Y-TZP groups (778 MPa, 870 MPa). It should be noted that the Katana ML zirconia has a multilayer shade gradient incorporated into the material, whereas the Lava Plus zirconia is uniform and stained prior to sintering. Despite the differences in coloring technique of the 3Y-TZP groups, there was no statistical differences found between the FS of the 3Y-TZP groups in this study.

The flexural fatigue strength was material dependent, but showed similar distribution as the FS decreased with the increase in the cubic phase contained in the materials. This was especially true for the Katana products, however, the exception to this finding was the 5Y-PSZ Lava Esthetic group which showed a higher flexural fatigue strength than 4Y-PSZ Katana STML group. The 5Y-PSZ Lava Esthetic group showed statistically similar FS to 4Y-PSZ Katana STML and 5Y-PSZ Katana UTML groups and statistically similar flexural fatigue strength to the 4Y-PSZ Katana STML group. However, testing showed strength degradation of the 5Y-PSZ Lava Esthetic percentages equal to those of the 3Y-TZP Katana ML and Lava Plus groups. It is unclear why this group diverged from the trend, but interestingly the manufacturers of Lava Esthetic report the highest biaxial FS of all the cubic zirconia materials currently available.[14] The data in this study did not substantiate this claim and found the 3-point bend FS to have no statistical difference to the other 5Y-PSZ group tested. However, the flexural fatigue strength did result in a higher value than that found in the other cubic containing groups. Obviously, between companies there is a difference in material chemical composition and sintering process. These proprietary formulas and fabrication methods have been shown to affect the material properties of zirconia.[19] Regarding the 3Y-TZP materials, the significant difference in flexural fatigue strength between

Katana ML and Lava Plus groups may be associated with the shaded layering of the Katana ML zirconia which may have decreased strength properties between layers. Or, it may simply be the difference in proprietary formulation of these products.

All tested ceramic materials showed significant strength degradation after 6,000 loading cycles. The severity of the degradation shows a high dependency on the differences in the materials' microstructure which can explain the observed variances. As discussed, the higher content of ytrria stabilizes the zirconia materials with a greater amount of cubic crystals in the microstructure which eliminates the transformation toughening mechanism. The lack of this mechanism is the primary factor in decreased FS and increased FS degradation of these materials.[20] The higher grain size found in the cubic containing zirconia materials may also contribute to the decreased FS and flexural fatigue strength values. Larger grain sizes can contribute to lower mechanical behaviors under static and fatigue assessments as smaller grain sizes require a higher applied stress to induce fracture. Smaller grains can limit the size of the dislocations on the crystal grain boundaries, thus increasing mechanical properties.[16,21,22]

Based on the FS values, ISO standard 6872-2015 would grade the 5Y-PSZ groups as class 4 materials to be utilized as single-unit anterior or posterior prostheses or as a 3-unit anterior prosthesis.[23] After fatigue testing, Katana UTML, would be re-classified as a class 2 ceramic, with recommendation to be utilized as an adhesively cemented anterior or posterior single-unit crown owing to its significant decrease in strength. The 4Y-PSZ Katana STML, based on FS values, would be categorized as a class 5 ceramic material, able to be utilized as a substructure ceramic for three unit prostheses involving molar restoration. However, after fatigue degradation, the flexural fatigue strength value found in this study would reclassify it to a class 4 ceramic material, making 4Y-PSZ Katana STML not recommended for a substructure ceramic for a three-unit prosthesis not involving molar restoration. This is contrary to manufacturer's recommendations. As a result of fatigue testing and expected increased degradation in the cubic containing products, care must be taken with the utilization of these cubic-containing materials in multiunit fixed dental prostheses.

Limited studies have looked at fatigue degradation of cubic containing materials. Pereira et al. performed biaxial flexural fatigue testing using a step stress fatigue approach for Katana ML, Katana STML, and Katana UTML.[16] Similar degradation percentages were found for Katana ML (32%, 31%), but significantly lower were found for Katana STML (27%, 43%) and Katana UTML (36%, 51%). Different testing methods may account for differences in results, but overall degradation should be expected, and in weaker materials, may have a more profound effect on the chosen clinical application.

The thickness of a restoration can be significant for both translucent and mechanical properties of ceramics. In this study, the data is related to a specimen thickness of 1.3 mm and is not bonded to a substrate. A thinner specimen could alter the performance of each zirconia material.[24] Further, bonding to a substrate would likely result in higher stresses to fracture, as shown by Campos et al.[24,25]

Conclusion

Based on our findings, the amount of strength degradation in zirconia materials was material dependent, with the 4Y-PSZ or 5Y-PSZ cubic containing zirconia materials demonstrating greater or similar strength degradation compared to the primarily tetragonal 3Y-TZP materials.

Disclaimer

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