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# **Effect of repeated post-sintering processes on the flexural strength of various multi-layered monolithic zirconia**

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## ABSTRACT

The aim of this study was to compare the flexural strength and flexural modulus of various multi-layered zirconia after being sintered and then undergoing multiple post-sintering processes. The study used three types of multilayered zirconia (Noritake Katana ML – 3Y, STML – 4Y, and UTML – 5Y). Pre-sintered zirconia pucks were milled using a 5-axis mill (MC X5) into bars (1.3x4x18mm). 80 bars were milled for each type of zirconia, then divided into 4 groups (n=20). All bars were sintered using a sintering furnace (Sirona InFire HTC Speed). After sintering, all samples were polished using P600 grit sandpaper. In the three post-sintered groups, samples underwent the following process 1, 2, or 3 times: air abrasion with aluminum oxide followed by application of glaze (Cerabien ZR glaze), and post-sintering firing using a ceramic furnace (VITA VACUMAT 6000M). All samples were subjected to a 3-point bend test using a universal testing machine (Alliance RT/5). Data was tested for normality using a Shapiro-Wilk test. A non-parametric Kruskal-Wallis test was used and comparisons for all pairs were done using Steel-Dwass method. There was a significant difference in the flexural strength of Katana ML Zirconia when comparing the samples that were sintered and the samples that underwent multiple post-sintering processes. There was no significant difference in the Katana STML Zirconia among the different groups. There was a significant difference in the Katana UTML

**Keywords:** *Flexural Strength, Post-Sinter, Sinter, Yttria, Zirconia*

## INTRODUCTION

The demand in dentistry for an improvement in esthetics as well as metal free materials has given way to ceramic restorations. (1) Conventional glass ceramics are known to be brittle, however there has been development of ceramics with improved strength that have been proven to be able to withstand the forces of mastication. (2) Among such ceramics is zirconia. Zirconia is a crystalline dioxide of zirconium. It has a color similar to tooth structure, yet the mechanical properties of metals, such as the thermal expansion, the elastic modulus, and high flexural strength making it a suitable definitive dental restoration. (5,6) Zirconia is present in three forms to include monoclinic, cubic, and tetragonal. In dentistry, yttria ( $Y_2O_3$ ) is added to the zirconia to stabilize it in its tetragonal form and is termed yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP). (2,4) Y-TZP has become popular in dentistry due to its biocompatibility, high strength, and the capability of transformation toughening; a phenomenon in which the material can minimize crack propagation by the material transitioning from its tetragonal to monoclinic form. (4,2,3,5).

3Y-TZP began as the most used form of zirconia in the dental field due to its high strength. However, having a high opacity resulted in it lacking in the ability to fulfill esthetic demands. (8) Increasing the content of the yttrium oxide stabilized more zirconia grains in the cubic phase, which increases the translucency of the material. However, this also drastically decreases the mechanical properties of the material. (8,10) In order to achieve the translucency desired yet maintain the mechanical components, 4Y-ZP was developed. (10)

96.2% of single-unit crowns are deemed clinically acceptable at delivery. (7) However, there are still 3.8% of crowns clinicians deem as needing adjustments, such as staining or adding a contact that require additional post-sintering firing processes before delivery (7).

Multiple post-sintering processes could influence the structural integrity of zirconia restorations. This study will help clinicians decide when, and if a new restoration should be re-fabricated (9).

The purpose of this article is to determine the effect of flexural strength and flexural modulus of zirconia restorations after they have been fully sintered and then endured numerous rounds of a post-sintering processes.

The hypothesis is that the flexural modulus and the flexural strength of the zirconia will perform differently after undergoing a post-sintering process three times. The null hypothesis is that there will be no difference in the flexural modulus and the flexural strength of the zirconia after undergoing a post-sintering process three times.

## **MATERIALS AND METHODS**

Three multi-layered zirconia with different yttria content were tested in this study: 3Y-TZP (Katana ML, Kuraray Noritake, Tokyo, Japan), 4Y-ZP (Katana STML, Kuraray Noritake, Tokyo, Japan), and 5Y-ZP (Katana UTML, Kuraray Noritake, Tokyo, Japan). (Table 1). Rectangular bars were digitally designed using a computer-aided design software (Autodesk Meshmixer, Autodesk Inc., San Rafael, CA) and exported in standard tessellation language (STL) format. 80 rectangular bars (1.3x4x18mm) for each type of zirconia were milled using a 5-axis milled (MCX5, Dentsply Sirona, City, State). Bars were removed from the pucks and sprues removed using a fine diamond bur. Samples were sintered using a sintering furnace (inFire HTC Speed, Dentsply Sirona, Charlotte, NC) following the manufacturer's instructions. The sintering process increased 10°C/min until reaching 1540°C and was held at this temperature for 2.5 hours. After being held for 2.5 hours, the temperature was decreased by 5°C/min until reaching room temperature. After sintering, the samples were polished using 600

grit sandpaper, 20 swipes per surface.

The samples were separated, air abraded with aluminum oxide 50 $\mu$ m at 2 bar (material, micron, pressure), steam cleaned, and glazed with Cerabien ZR (Noritake Kuraray, Tokyo, Japan) before undergoing the post-sintering process. The ceramic furnace (VITA VACUMAT 6000M, Vita North America, Yorba Linda, CA) was used to complete the post-sintering following the manufacturer's guidelines. Each sample reached a temperature of 750°C with a heating rate of 45°C/min and held for 1 minute. Vacuum cycles were held between 500°C and 7500°C. The cooling process began at 400°C and was held for 3 minutes before continuing to cool. The final sample groups included those that were sintered (S), or those having endured a post-sintering process once (PS1), twice (PS2), or three times (PS3).

The flexural strength was measured using a 3-point bending fixture (15mm span) mounted in the universal testing machine (Alliance RT/5, Material Testing Systems Corporation, Eden Prairie, MN) with a crosshead speed of 0.5 mm/min and recorded using TW Elite Software (Material Testing Systems Corporation, Eden Prairie, MN). (10) The flexural modulus was calculated by taking the value of the stress divided by the strain.

Table 1: Test Material Information

Material	Manufacturer	Chemical Composition
Katana Zirconia Multi-layered Zirconia (ML)	Kuraray Noritake Dental Inc	3 mol % Y2O3 Tetragonal Zirconia Polycrystals
Katana Zirconia Super Translucent Multi-layered Zirconia (STML)	Kuraray Noritake Dental Inc	4 mol % Y2O3 Zirconia Polycrystals
Katana Zirconia Ultra Translucent Multi-layered Zirconia (UTML)	Kuraray Noritake Dental Inc	5 mol % Y2O3 Zirconia Polycrystals

## RESULTS

To analyze the flexural strength and the flexural modulus for normality, a Shapiro-Wilks test was performed. The flexural modulus was determined to be normal among all groups. Regarding flexural strength, it was determined the data for ML and UTML were normally distributed however the STML group was not normally distributed. A Kruskal Wallis test was conducted to determine if there are statistically significant differences among the groups. Looking at the flexural modulus, there was a statistical difference amongst all groups: ML ( $p = <0.0001$ ), STML ( $p = <0.001$ ), and UTML ( $p = <0.0001$ ). (Figure 1) The flexural strength was determined there was not a significant difference among the STML ( $p = 0.2820$ ) but a statistical significance was found among the ML ( $p = <0.0001$ ) and UTML ( $p = 0.0094$ ) groups. (Figure 2) The Steel-Dwass method was used to examine the pair comparisons of each group. With the flexural modulus the following was found; in the ML group there was a statistical significance between the S and PS1 ( $p = <0.0001$ ), PS2 ( $p = <0.0001$ ) and PS3 ( $<0.0001$ ), there were also significant differences among all the ML post-sintered groups. The STML group was found have statistical differences between the S and PS1 ( $p = <0.0001$ ), PS2 ( $p = <0.0001$ ), and PS3 ( $p = <0.0001$ ). The post-sintered STML groups were all found to be statistically significant from one another. The UTML was found to have statistical differences among the S and PS1 ( $p = <0.0001$ ), PS2 ( $p = <0.0001$ ), and PS3 ( $p = <0.0001$ ). Within the post-sintered UTML groups, the only significance found was between the PS1 and PS3 ( $p = 0.0026$ ). (Figure 1) The analysis of the flexural strength found there were no significant differences among the STML group. Among the ML group there was a statistical difference between the S and the PS1 ( $p = 0.0032$ ), PS2 ( $p = 0.0082$ ), and PS3 ( $p=0.0002$ ). Among the UTML there was a statistical difference between the PS1 and PS3 ( $p = 0.0154$ ) and the PS2 and PS3 ( $p = 0.0154$ ). (Figure 2)

The Weibull modulus, character strength, and percent difference of the character strength was calculated for each material and the different post-sintering processes. (Table 2,3,4) The UTML PS3 was seen to have the highest Weibull modulus. The percent difference in strength can be seen in (Figure 3). ML is seen to have a decrease in the percent difference of strength when comparing the sintered versus the post-sintered.

Table 2: Calculated Weibull Modulus and Character Strength for ML

Material	N	Weibull Modulus	Characteristic Strength (MPa)
S ML	19	8.85	671.43
PS-1 ML	19	9.01	555.29
PS-2 ML	19	8.58	565.69
PS-3-ML	19	6.37	516.37

Table 3: Calculated Weibull Modulus and Character Strength for STML

Material	N	Weibull Modulus	Characteristic Strength (MPa)
S STML	19	8.74	467.77
PS-1 STML	19	6.87	509.84
PS-2 STML	19	7.16	462.30
PS-3-STML	19	7.21	485.93

Table 4: Calculated Weibull Modulus and Character Strength for UTML

Material	N	Weibull Modulus	Characteristic Strength (MPa)
S ML	19	8.85	671.43
PS-1 ML	19	9.01	555.29
PS-2 ML	19	8.58	565.69
PS-3-ML	19	6.37	516.37

Figure 1:

Columns per group with the same case letter are not significantly different than each other ( $p > 0.05$ ).

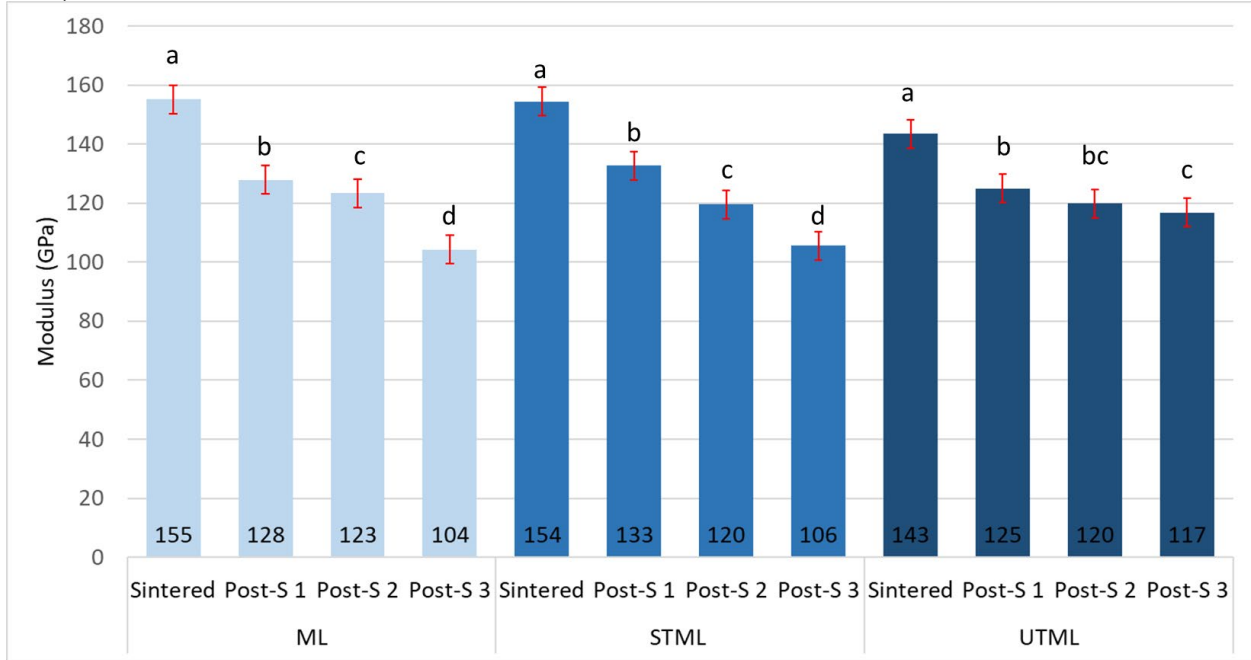


Figure 2:

Columns per group with the same case letter are not significantly different than each other ( $p > 0.05$ ).

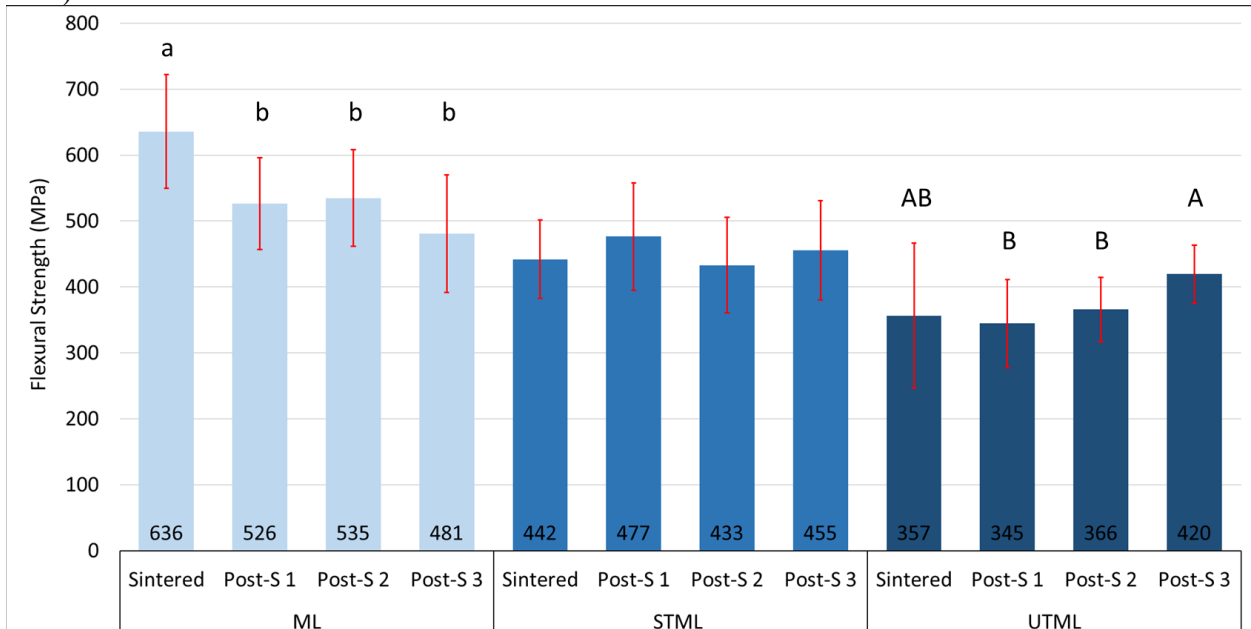
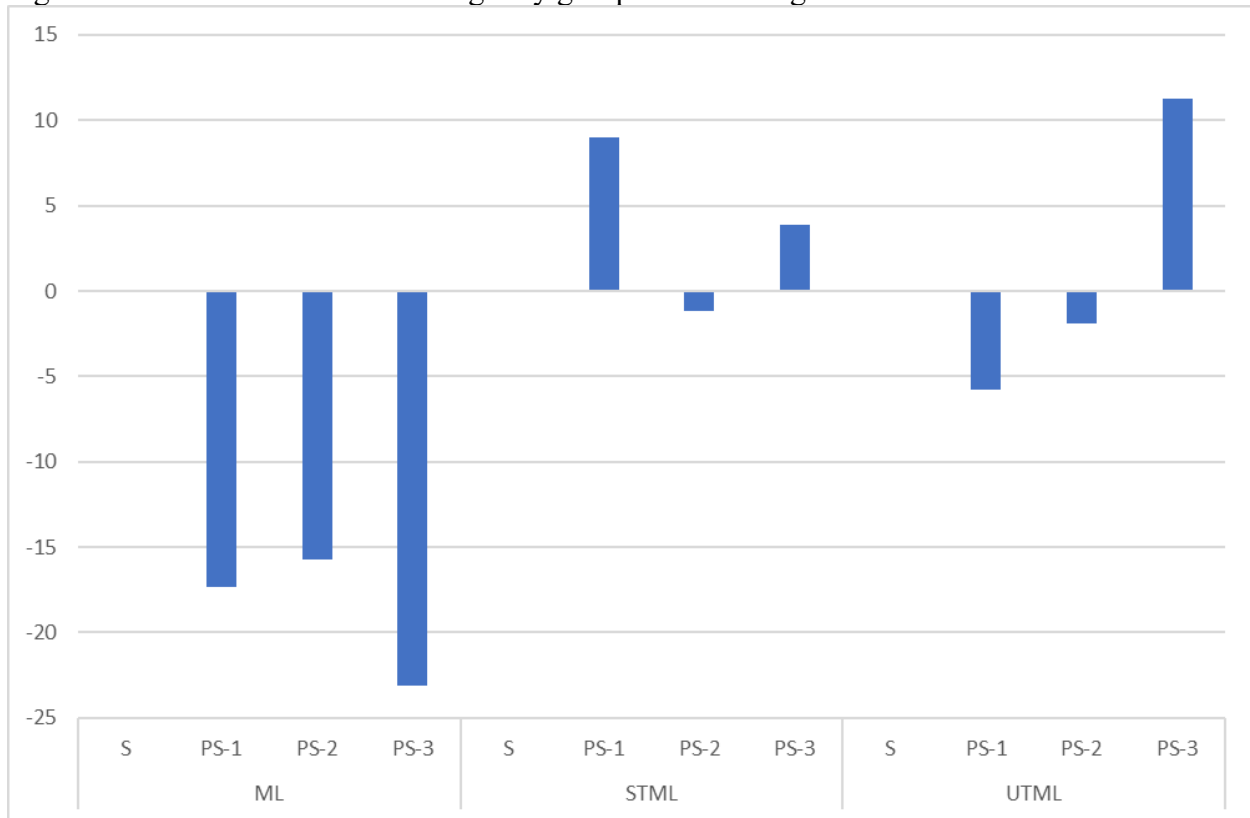


Figure 3: Percent difference in strength by group and sintering time



## DISCUSSION

This in vitro study investigated the flexural strength of Katana ML, STML, and UTML after being sintered, and then following three post-sintering processes. The null hypothesis was rejected for Katana ML, STML, and UTML because there was a significant difference in the flexural modulus amongst all groups. The null hypothesis was rejected for the Katana ML and because the results showed that there were significant differences in the flexural strength among the specimen that were sintered and the specimen that had undergone the post-sintering process. The null hypothesis was accepted for the STML and UTML groups which showed no statistical difference among the sintered and post-sintered groups.

Multi-layered zirconia has entered the market and there are several manufacturers with complete products. In this study, Katana ML, STML, and UTML was chosen. This multi-layered zirconia has the same distribution of yttria content in each puck. It is the shade gradient that is multi-layered. Other multi-layered zirconia in the market has a different distribution of Y-TZP in the same puck.

The flexural strength of the ML group was noted to have a significance difference between the sintered, PS1, PS2, PS3. This could be due to the alumina content in the 3Y-TZP. The alumina content exists to help in stabilization of the tetragonal phase of zirconia. (11) In the second generation of zirconia, alumina was decreased from 0.25wt% to 0.05wt% which allowed for more translucency but also resulted in the material being more susceptible to low-temperature degradation. (11). Another explanation is provided in study by Song (15). Song suggested that air-borne particle abrasion could be used to increase flexural strength because of the promotion of phase transformation, however, strength degradation could result from excessive amounts of transformed monoclinic phase and slow crack growth. (15,16)

Although not determined to be significant, there was a slight increase noted in the flexural strength of the UTML as it endured multiple most sintering processes. This is different than findings in previous studies. (14) McLaren studied the influence of surface treatments on the strength of translucent zirconia's and found that airborne-particle abrasion with alumina had strengthening effects on the 4 mol% zirconia but a weakening effect on the 5 mol% zirconia. (14) The difference in findings of McLaren's versus this study could be attributed to the fact that post-treatment with the air-abrasion aluminum oxide (50 $\mu$ m, 2 bar).

The flexural strength values found in this study were lower than what has been recorded by the manufacturer and in other studies. (11,13) While a study by Kwon et al. (11) recorded the

flexural strength values of the Katana HTML as being  $1194 \pm 111$  MPa. The difference in these values could be due to the measurements of the specimen being larger than those used in this study, as well as variations in sintering furnaces, surface treatments, etc. However, this study did find that the flexural strength of the material decreases from Katana ML to Katana STML, to Katana UTML. This remains consistent with findings of the manufacturer and other studies (12,13).

## **CONCLUSION**

Within the limitations of this study, the results show that there is a significant difference in the flexural modulus of all the materials tested. The flexural strength of ML when subjected to post-sintering processes did have a significant difference. There was not a statistical difference noted in the flexural strength of STML or UTML when subjected to post-sintering processes.

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