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Effect of Different Surface Treatments on Biaxial Flexural Strength of Yttria-stabilized Tetragonal Zirconia Polycrystal

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ABSTRACT

Introduction: Ceramics are widely applied in dentistry owing to their excellent mechanical and physical attributes. The most popular ceramics are Lava™, KaVo Everest, and Cercon. However, it is unclear whether or not a different surface treatment along with low-temperature aging and mechanical loading (ML) affects the physical properties of computer-aided design (CAD)/computer-aided manufacturing (CAM)-machined yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic.

Aim: The objective of this research was to assess the impact of various surface treatments as air-particle abrasion, ML, low-temperature degradation (LTD), and their cumulative effects on biaxial flexural properties of Y-TZP.

Materials and methods: Totally, 50 specimens were fabricated by CAD–CAM machining from Cercon® and divided into five groups following different surface treatments as control (C), air-particle abrasion (Si), ML, LTD, and cumulative treatment (CT) group. Results were investigated by two-way analysis of variance (ANOVA) and Tukey honest significant difference (HSD) test.

Results: The highest biaxial flexural strength was observed in the Si group (950.2 ± 126.7 MPa), followed by the LTD group (861.3 ± 166.8 MPa), CT group (851.2 ± 126.5 MPa), and the least with ML (820 ± 110 MPa). A significant difference was observed in the two-way ANOVA test. X-ray diffraction (XRD) analysis showed that the control group consists of 100% tetragonal zirconia and the maximum amount of monoclinic phase was obtained after LTD.

Conclusion: No negative effect on biaxial flexural strength was observed; indeed, it increases the biaxial strength. Hence, these surface treatments can be done in routine clinical practice to improve the performance of ceramic restoration.

Keywords: Biaxial flexural strength, Computer-aided design–computer-aided manufacturing, Phase transformation.

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INTRODUCTION

The uses of ceramics are not new in dentistry. Ceramics, especially Y-TZP, are widely used in dentistry, especially in the field of prosthodontics and restorative dentistry because of their excellent mechanical, physical, and optical properties.[1] However, during certain procedures such as CAD–CAM machining and grinding, use of diamonds, stone, and abrasives and during sterilization procedures resulted in the development of surface flaws,[2] leading to stress concentration at specific sites.[3]

The Y-TZP ceramics demonstrated superior strength due to its phase transformation phenomenon. During this phenomenon, there is approximately a 4% increase in volume due to tetragonal to monoclinic transformation.[4] This transformation may also occur during air-particle abrasion, grinding, and temperature changing during autoclaving as low thermal degradation.[5,6]

There is a wide variety of CAD–CAM zirconia materials available in the market, which have shown excellent physical and mechanical properties when compared with high alumina ceramics.[7] However, it is unclear whether or not a different surface treatment along with low-temperature aging and ML affects the physical properties of CAD–CAM-machined Y-TZP ceramic.

Hence, the goal of this study is to assess and compare the influence of different surface treatments and their accumulative effect on biaxial properties and phase transformation of Y-TZP ceramics.

MATERIALS AND METHODS

Fifty disk-shaped specimens of Cercon® base (Degudent, Hanau, Germany) (Fig. 1) were prepared as per International Organization for Standardization (ISO) 6872 1995 standard. The standard described that a test piece should have a minimal thickness of 1.2 ± 2 mm and a diameter of 12 to 16 mm. The specimens were initially milled in large dimensions to compensate for the shrinkage occurring during sintering. In the previous surveys, it was noted at around 25% for Cercon. The specimens were then sintered in a sintering oven at 1,350°C for about 1.5 hours as per the manufacturer recommendation. The materials used in this study are presented in Table 1.

The groups are organized as follows:

Control group (C): Consisted of CAD–CAM-machined specimens. Not subjected to any treatment after fabrication.

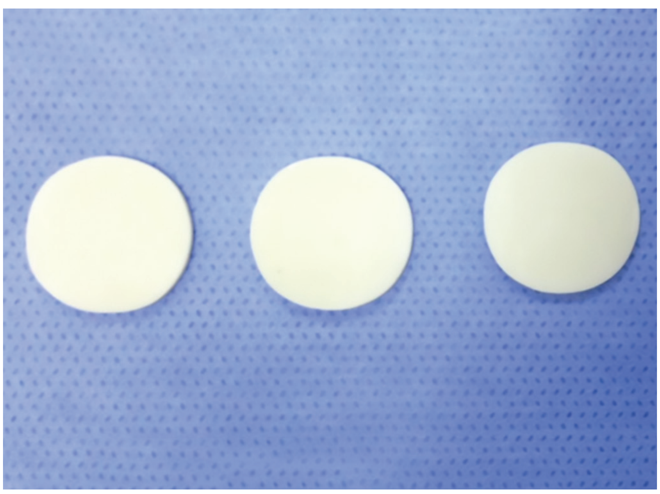


Fig. 1: Disk-shaped specimens

Table 1: Brand, composition, and manufacturers of materials used in this study

Brand	*Composition*	*Manufacturers of materials*
Cercon® base	ZrO_2 (92 vol%), Y_2O (35 vol%), HfO_2 (2 vol%)	DeguDent, Hanau, Germany
CoJet™ Sand	30 μm silica-coated Al_2O_3 particles	3M ESPE

Air-particle abrasion group (Si): Specimens were sandblasted with 30 μm silica-coated alumina particles (CoJet™ sand) at 0.28 mm pressure. After sandblasting, all the specimens were cleaned in the ultrasonic cleaner for 10 minutes.

ML group: A cyclic load of 10,000 cycles was applied centrally to the specimen in 37°C water at 2 Hz using the load between a minimum and a maximum force from 20 to 250 N. During the loading, the maximum force was set to mimic occlusal loading in the posterior tooth region, which was approximately 25% of mean biaxial flexural strength.

LTD group: Specimens were autoclaved at 127°C at 1.5 bar pressure for 12 hours, which induces LTD in zirconia.

CT group: Specimens were subject to surface treatment, ML, and LTD.

Density measurements: It was performed on each sintered specimen using the Archimedes principle, calculated using the equation

$$\rho = \text{Actual weight/actual-suspended} \times \rho_w$$

where ρ = Density of the sample (gm/cm^3) and ρ_w = Density of water (gm/cm^3).

Biaxial Flexural Strength

The testing was executed as per ISO 6872 spec. Instron 8871 Servohydraulic system (Instron®, US) was employed (Fig. 2). A jig was constructed to contain the specimen. It was designed with a support circle of 11 mm diameter, and three steel balls were positioned at 120° angles. A loading pin was used of length 2 mm and a diameter of 1.5 mm. Samples were placed on the supporting balls and then loaded with indenter at a crosshead speed of 1 mm/min until a fracture occurred. Failure load was recorded using graph data manager software. Biaxial

Fig. 2: Biaxial flexural strength testing using universal testing machine

Table 2: Comparison of biaxial flexural strength and Weibull statistics of Cercon® group specimens

Group	*Mean biaxial flexural strength in MPa (SD)*	*Mean standard error (SE)*	*Characteristic strength (σ) (MPa)*	*95% confidence intervals for characteristic strength (σ)*	*Weibull modulus (m)*	*95% confidence intervals for Weibull modulus*
Control (C)	827.9 ± 115	5.140	852.95	808.07–900.18	7.9	6.6–9.5
Air-particle Abrasion (Si)	950.2 ± 126.7	4.794	1004.9	963.0–1048.7	8.8	6.6–11.5
ML	820 ± 110	4.283	850	830–870	7.9	6.2–10.1
LTD	861.3 ± 166.8	5.074	1024.8	997.28–1053.1	8.2	6.2–10.8
CT	851.2 ± 126.5	5.102	1162.0	1102.4–1224.6	5.6	4.3–7.5

SD: Standard deviation

flexural strength (MPa) was calculated using following formula as per ISO 6872 1995 standard.

$$\sigma = \frac{-0.2387\,P(X-Y)}{d2}$$

where σ is biaxial flexural strength, P = max load, L = length (mm), and d = specimen thickness.

X-ray Diffraction Analysis

The XRD analysis was carried out to determine the crystalline phase. Five specimens were taken from each group for the analysis. The XRD data were obtained with a θ-20 diffractometer r (Models: Rigaku Ultima IV and JEOL JDX 3530) using Cu–Kα radiation. Garvie and Nicholson's method was applied to determine the monoclinic phase in samples. It is stated in terms of percentage of tetragonal phase that was transformed to monoclinic phase.

$$X_m = \frac{(I_{m1} + I_{m2})}{(I_{m1} + I_{m2} + I_t)}$$

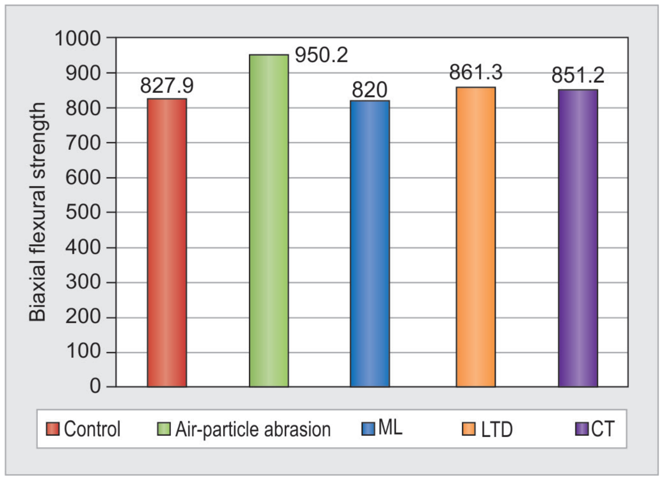
where I = intensity at angular position 20°.

RESULTS

Table 2 depicts mean biaxial flexural strength plus respective standard error of the mean of Cercon specimen. There was an increase in biaxial strength of the Cercon air abrasion group (Si), LTD group, and CT group except for ML group where biaxial flexural strength is decreased as compared with control group. In Graph 1, biaxial flexural strength is graphically represented. Highly significant differences ($p = 0.000$) were found between the control and test group in two-way ANOVA (Table 3) analysis. Tukey HSD (Table 4) was carried out further to determine any significant difference among the groups. Statistically significant ($p < 0.05$) was observed between C and Si group and LTD group specimens. On the contrary, there was no significant difference ($p \geq 0.05$) found between C and ML groups and C and CT group specimens.

Weibull analysis was borne away to see the variability of flexural strength values. The formula used is as follows:

$$P(\sigma) = 1 - \exp[-(\sigma/\sigma 0)m]$$



Graph 1: Comparison of biaxial flexural strength of Cercon® specimens after different surface treatments

Table 3: Two-way ANOVA results

	Sum of squares	*Df*	*Mean square*	*f-value*	*p-value*
Between groups	111796.120	4	27949.030	116.932	0
Within groups	10755.900	45	239.020		
Total	122552.020	49			

Df: Degree of freedom

Table 4: Tukey HSD test results

Variable (I)	*Variable (J)*	*Mean difference (I-J)*	*Standard error*	*p-value*	*95% confidence interval*	
					Lower bound	*Upper bound*
Control	ST	−127.200*	6.914	0*	−146.85	−107.55
	ML	2.900	6.914	0.993	−16.75	22.55
	LTD	−38.300*	6.914	0*	−57.95	−18.65
	CT	−27.700	6.914	0.052	−47.35	−8.05

*The mean difference is significant at the 0.05 level

Table 5: Relative amount of monoclinic zirconia (%) of the tested groups

Groups	*Monoclinic phase (%)*
Control (C)	0
Air-particle abrasion (Si)	8
ML	6.2
LTD	26.43
CT	12.58

where P = Probability of failure, r = Strength at given P, σ_0 = characteristic parameter and m = Weibull modulus.

Table 5 shows that there was no monoclinic phase present in the control group; however, another group showed a variable amount of m phase. The variation was observed from 0 to 27%. Si group and the ML group showed 8 and 6.2% m phase respectively. The LTD and the CT groups showed variations in the m phase from 26.43 to 12.58%.

DISCUSSION

This study was performed to determine the outcome of various surface treatments and their CT effect on biaxial flexural strength and phase transformation. Performance of brittle material such as ceramics can be determined by evaluating strength, which is described as ultimate strength required to fracture or cause plastic deformation of ceramics.[8] Different methods are discussed in the literature to measure flexural strength as a 3-point test, 4-point test, or biaxial flexural test. Among these tests, biaxial flexural strength test is widely recognized.[9,10] As the maximum tensile stress occur in central loading area.[11]

As observed in previous studies, airborne-particle abrasion during sandblasting or polishing procedure may create internal flaws. The results of this study indicated that after airborne particle abrasion with 30 μ silica-coated alumina particle, there was an improvement of biaxial flexural strength of specimens. This can be explained by the fact that tetragonal to monoclinic phase transformation creates a layer of compressive stress that counteracted the degradation of strength by surface flaws. However, the surface flaws created by sandblasting have not exceeded the compressive layer thickness, which could have resulted in a decrease in strength rather than an increase in strength.[12]

In this study, air-particle abrasion resulted in approximately 8% monoclinic to tetragonal phase transformation. Studies in the past have shown similar results, where the authors have found that the improvement of strength was because of an increase in monoclinic phase percentage.[13-16] A study conducted by Zhang et al[17] concluded that increase in strength of Cojet sandblasted specimens was attributed to their smaller size as well as their soft and round configuration. Curtis et al[18] reported similar behavior with 25 μm Al_2O_3 particles.

The results in this present study showed that the mechanical cyclic loading at 10,000 cycles in water, using a force of 250 N, did not significantly ($p \geq 0.05$) affect the biaxial flexural strength of specimens. These findings are in agreement with a study conducted by Itinoche.[19] However, the ceramics used were different from the current study. Curtis et al[20] evaluated the effect of biaxial flexural strength of zirconia after subjecting the specimens under various forces varying from 500 to 800 N for 2,000 cycles and found that the strength of samples was not deteriorated.

The present study's LTD has not shown any significant reduction in biaxial flexural strength of specimens. Previous studies have shown that autoclaving at 134°C for 1 hour has the same effect of 3 to 4 years of aging.[21,22] Therefore, accelerated aging test was performed with autoclaving at 134°C for 10 hours under 0.2 MPa pressure, which induces LTD in zirconia.[22] Similar findings have been reported by Pröbster and Diehl.[23] Another study reported that there is no statistically significant difference in flexural strength of zirconia aged at 37°C for 1 year.[24]

Shimizu et al[25] carried out an experiment to determine the effect of temperature on the specimen flexural strength after placing them in saline solution for 3 years and distilled water at 121°C for 2000 hours. His investigation confirmed that there was no significant change in flexural strength in ceramic specimens even after such long LTD treatment.[25]

An interesting finding of this present study was that the biaxial flexural strength of the CT specimens increased as compared with the control (C) and ML but less as compared with air-particle abrasion group (Si) and LTD group. This can be explained by the fact that compressive force generated by tetragonal to monoclinic transformation has overcome the deteriorating effect of different surface treatment. The same observation was reported by Guazzato et al[26] and Kosmac et al[12] in their studies, where they had observed the effect of air-particle abrasion and aging.

The "m" values observed in this study were in the range of 5 to 15. These are the normal values quoted for dental ceramics.[14] However, previous studies have shown higher values.[3,27-29] Few groups demonstrated less Weibull modulus as compared with control group. This signifies that surface treatment might have affected the reliability of clinical performance of ceramics. Weibull values were demonstrated in Table 2. However, larger Weibull values represent that there are fewer critical flaws and indicate a smaller error in judgment of clinical strength.[14]

Characteristic properties of zirconia are attributed to its tetragonal to monoclinic phase conversion. The observations of the present study are in agreement with previous studies, where the C group consisted of 100% tetragonal zirconia.[3,12,30]

The Y-TPZ remains stable in the tetragonal state between 1,145°C and below room temperature. Different surface treatments such as air-particle abrasion or ML lead to phase transformation.[3,12,14,31,32] This could be explained by the fact that when they are exposed to stress, change in crystal cell structure occurs due to tetragonal to monoclinic transformation.[33,34]

Table 5 depicts the relative percentage of monoclinic content of treated specimens. The greatest quantity of monoclinic phase was detected following LTD in the present study. Similar results were obtained in previous studies by Kosmac et al[3] and de Kler et al.[35] Many of the previous studies have shown no monoclinic content in the control group.

Limitation

One of the major shortcomings of this study was that it does not mimic the clinical condition exactly, which may produce different results due to the presence of saliva and pH changes. The long-term evaluation of the zirconia restoration under clinical condition should also be studied, especially for long span.

CONCLUSION

The conclusions are as follows:

- The highest biaxial flexural strength was observed in the air-particle abrasion group, followed by LTD group, cumulative group, and the least with the ML group.
- The ML under 250 N forces reduces biaxial flexural strength which was approximately 25% of mean biaxial flexural strength. However, it was not statistically significant.
- A 100% tetragonal zirconia was observed in the control group, and the greatest amount of monoclinic percentage was observed after LTD treatment.

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