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Effects of various surface treatments on the biaxial flexural properties of yttria-stabilized zirconia ceramics

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Abstract

Aim: The aim of the present study was to evaluate and compare the influence of different surface treatments and their cumulative effects on the biaxial flexural properties and phase transformation of yttria-stabilized zirconia ceramics.

Materials and Methods: A total of fifty specimens were fabricated by computer-aided design/computer-aided manufacturing machining from Cercon®. The samples were divided into five groups following different surface treatments as control (C), air particle abrasion (Si), mechanical loading (ML), low-temperature degradation (LTD), and cumulative treatment (CT) groups.

Statistical Analysis Used: The results were analyzed by two-way ANOVA and Tukey's honestly significant difference (HSD) test. Two-way ANOVA was used to find significance between the test and the control groups. Tukey's HSD test was carried out to determine any significant difference among the groups.

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 the ML group (820 ± 110 MPa). Significant difference was observed in two-way ANOVA test. Tukey's HSD test showed that there was a significant difference ($P \leq 0.05$) between the C and Si groups and C and LTD groups; however, no significant difference was observed ($P \geq 0.05$) between the C and ML groups and C and CT groups. X-ray diffraction analysis showed that the control group consisted of 100% tetragonal zirconia while the maximum amount of monoclinic phase was obtained after the LTD treatment.

Conclusions: Air particle abrasion with CoJet Sand, LTD, and CTs had no negative impact on biaxial flexural strength indeed it increased the biaxial flexural strength. Hence, these surface treatments can be done in routine clinical practice to improve the performance of ceramic restorations.

Key words: Biaxial flexural strength, computer-aided design/computer-aided manufacturing, phase transformation, surface treatments, zirconia

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Introduction

The use of ceramics is not new in dentistry. Ceramics are widely used in dentistry, especially, in the field of prosthodontics and restorative dentistry because of their excellent mechanical, physical, and esthetic properties. Due to their excellent mechanical properties, there is even higher use of zirconia in dentistry, especially, yttria-stabilized zirconia (Y-TZP) ceramics.[1] However, certain procedures such as computer-aided design/computer-aided manufacturing (CAD-CAM) machining and various other procedures including sterilization result in development of surface flaws that lead to stress concentration at specific sites.[1,2] The abrasives regularly used in dental applications include silica-coated alumina particles of different sizes. However, concerns have been raised following treatment with such abrasives. There are studies which have demonstrated the influence of these abrasives on the physical properties of zirconia.[3] Y-TZP ceramics demonstrate superior strength due to their phase transformation phenomenon. During this phenomenon, there is approximately 4% increase in volume due to tetragonal to monoclinic transformation.[4] This transformation may, also, occur during various procedures including air particle abrasion, mechanical loading (ML), and low-temperature degradation (LTD) during autoclaving and cumulative procedures during the fabrication of the prosthesis.[5,6] When zirconia restorations are subjected to heavy masticatory forces and thermal stresses, it leads to further deterioration of their strength due to crack propagation. Due to this reason, the evaluation of effects of different surface treatment and their cumulative effects becomes the need of the hour. There are a wide variety of CAD-CAM zirconia materials which are available in the market and which have shown excellent physical and mechanical properties when compared to high alumina ceramics.[7] Some such common examples include Lava™, KaVo Everest, and Cercon. However, it is unclear whether or not a different surface treatment along with low-temperature aging and ML together affects the physical properties of CAD-CAM machined Y-TZP ceramics. The aim of the present study was to evaluate and compare the influence of different surface treatments and their cumulative effects on the biaxial flexural properties and phase transformation of Y-TZP ceramics.

Materials and Methods

Fifty disc-shaped specimens of Cercon® base [Figure 1] (Degudent, Hanau, Germany) were prepared as per ISO 6872 1995 standards. The standard described that a test piece should have a minimum thickness of 1.2 (±2) mm and a diameter of 12–16 mm. The specimens were initially milled in large dimensions to compensate for the shrinkage occurring during sintering. In the previous studies, it was noted around 25% for Cercon. The specimens were, then, sintered in sintering oven at 1350°C for about 1.5 h as per the manufacturer recommendations. The materials used in the study are shown in Table 1. The specimens were divided into the following groups based on surface treatments:

Figure 1: Disc-shaped specimens

- Control group (C) - Consisted of CAD-CAM machined specimens. Not subjected to any treatment after fabrication
- Air particle abrasion (Si) group - Specimens were sandblasted with 30 μm silica-coated alumina particles (CoJet™ sand) at 0.28 mm pressure. After sand blasting, all the specimens were cleansed in an ultrasonic cleaner for 10 min
- ML group - A cyclic load of 10,000 cycles was applied centrally to the specimens in 37°C water at 2 Hz using with the load between a minimum and a maximum force from 20 to 250 N. During the loading phase, the maximum force was set to mimic occlusal loading in posterior teeth region which was approximately 25% of the mean biaxial flexural strength
- LTD group - Specimens were autoclaved at 127°C at 1.5 bar pressure for 12 h which induced degradation
- Cumulative treatment (CT) group- Specimens were subjected to air particle abrasion, ML, and LTD
- Density measurements- Density measurements were performed on each sintered specimen using the Archimedes' principle calculated using the following equation:
  ρ = actual weight/actual suspended × ρ_w
  Where, ρ = density of the sample (g/cm^3) while ρ_w = density of water (g/cm^3)
- Biaxial flexural strength measurement - BiAxial flexural strength measurement was performed as per ISO 6872 specifications. Instron 8871 Servo hydraulic system (Instron®, US) was used [Figure 2]. A jig was fabricated to hold the specimen. It was designed with a support circle of 11 mm diameter,

Table 1: Brands, composition, and manufacturers of materials used

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

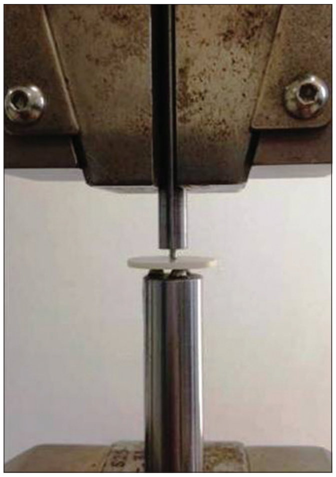

Figure 2: Biaxial flexural strength testing using universal testing machine

and three steel balls were positioned at 120° angles. A loading pin was used of length 2 mm and diameter 1.5 mm. Samples were placed on the supporting balls and then loaded with an indenter at a cross head speed of 1 mm/min until fracture occurred. Failure load was recorded using graph data manager software. Biaxial flexural strength (MPa) was calculated using the following formula as per ISO 6872 1995 standards:

$\sigma = -0.2387\, P\, (X\text{-}Y)/d_2$

Where, σ is biaxial flexural strength, *P* = maximum load, *L* = length (mm) while *d* = Specimen's thickness

- Weibull analysis – Weibull analysis was carried-out to determine the variability of flexural strength values. The formula used was:

  $P(\sigma) = 1\text{-}\exp(-[\sigma/\sigma_0]^m)$

  Where, *P* = Probability of failure, *r* = strength at a given *P*, σ_0 = characteristic parameter while m = Weibull modulus
- X-ray diffraction analysis (XRD analysis): XRD analysis was carried out to determine the crystalline phase. Five specimens were selected from each group for the analysis. XRD data were obtained with a θ-20 diffractometer (Models: Rigaku Ultima IV and JEOL JD × 3530) using cu-Kα radiation. Garvie and Nicholson's method was used to determine monoclinic phase in the samples. It was expressed in terms of percentage of the tetragonal phase that was transformed to monoclinic phase.

  $Xm = (I_{m1} + I_{m2})/(I_{m1} + I_{m2} + I_t)$

  Where, *I* = intensity at angular position 20°.

Statistical analysis used

Two-way ANOVA was used to find significance between the test and the control groups. Tukey's honestly significant difference (HSD) test was carried out to determine any significant difference among the groups.

Results

Table 2 depicts the mean biaxial flexural strength plus respective standard errors of mean of Cercon specimens. There was increase in the biaxial flexural strength of cercon air particle abrasion (Si) group, LTD group, and CT group except in the ML group where biaxial flexural strength was actually decreased as compared to the control group (C). Highly significant difference ($P = 0.000$) was found between the control and test groups in two-way ANOVA which was used to find significance between the test and the control groups [Table 3]. Tukey's HSD test was carried out, further, to determine any significant difference among the groups wherein a statistically significant difference ($P < 0.05$) was observed between C and Si and C and LTD group specimens. On the contrary, there was no significant difference ($P > 0.05$) was found between C and ML and C and CT group specimens [Table 4]. Weibull analysis was carried out to determine the variability of flexural strength values which showed that there was no monoclinic (m) phase present in the control group; however, other groups showed variable amounts of m phase. The variation was observed from 0% to 27%. Si and ML groups showed 8% and 6.2% m phase, respectively while LTD and CT groups showed variation in m phase from 26.43% to 12.58% [Table 5].

Discussion

The aim of the present study was to evaluate and compare the influence of different surface treatments and their cumulative effects on the biaxial flexural properties and phase transformation of Y-TZP ceramics. The performance of brittle materials such as ceramics can be determined by evaluating strength which is described as ultimate strength required to fracture or lead to plastic deformation of the physical structure.[8] There are different methods discussed in the literature to measure flexural strength as 3-point test, 4-point test, or biaxial flexural test. Among these, biaxial flexural strength test is widely recognized as the

Table 2: Comparison of biaxial flexural strength and Weibull Statistics of Cercon® Group specimens

Group	Mean biaxial flexural strength in MPa (SD)	Mean SE	Characteristic strength (σ) (MPa)	95% CI for characteristic strength (σ)	Weibull modulus (m)	95% CI 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	850.0	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

ML: Mechanical loading, LTD: Low-temperature loading, CT: Cumulative treatment, SE: Standard error, CI: Confidence interval, SD: Standard deviation

Table 3: Two-way ANOVA results

	Sum of squares	df	Mean square	*F*	*P*
Between groups	111,796.120	4	27,949.030	116.932	0.000*
Within groups	10,755.900	45	239.020		
Total	122,552.020	49			

*The mean difference is significant at $P<0.05$

maximum tensile stress occurs within the central loading areas.[9-11] As observed in the previous studies, air borne particle abrasion during sandblasting as well as polishing procedures creates internal flaws leading to decreased strength. The results of the present study, however, on the contrary, indicated that after air borne particle abrasion with 30 µm silica-coated alumina particles, there was an improvement in the biaxial flexural strength in the tested specimens. This can be explained by the fact that tetragonal to monoclinic phase transformations within the physical structure 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 decrease in strength rather than an increase.[12] In the present study, air particle abrasion resulted in approximately 8% monoclinic to tetragonal phase transformations. The studies in the past have shown similar results with a conclusion that the improvement in strength was because of an increase in monoclinic phase percentages.[13-16] A study conducted by Zhang *et al*. concluded that increase in strength of CoJet sand-blasted specimens was attributed to their smaller size as well as their soft and round configuration.[17] Curtis *et al*. reported similar behavior with 25 µm Al_2O_3 particles.[18] The results in the present study, also, showed that mechanical cyclic loading at 10,000 cycles in water using a force of 250 N did not significantly ($P > 0.05$) affect the biaxial flexural strength in the tested specimens. Similarly, a recent study investigated the flexural strength of In-Ceram Zirconia after fatigue and used a force of 50 N for 20,000 cycles. The results of the said study concluded no significant difference between before and after cycling flexural strength.[19] Furthermore, the materials used in the study conducted by Sobrinho *et al*. showed higher fatigue resistance than in the present study which might be explained because of the differences in the constitution of the said ceramics since In-Ceram Zirconia contains 35% zirconium oxide while Procera is a polycrystalline ceramic which would have higher fatigue resistance.[20] Curtis *et al*. evaluated the effect of biaxial flexural strength of zirconia after subjecting the specimens under 500, 700, and 800 N force for 2000 cycles and found that the strength of specimens was not deteriorated. Although the samples were tested to 100,000 cycles using an 80 N force, the biaxial flexural strength was still no different.[21] In the present study, LTD did not show any significant reduction in the biaxial flexural strength in the tested specimens. Numerous studies conducted in the past have shown that autoclaving at 134°C for around 1 h has a similar effect as 3–4 years of ageing.[22,23] Therefore, accelerated ageing test was performed with autoclaving at 134°C for around 10 h under 0.2 MPa pressure which induced degradation in zirconia.[23] Similar findings have been reported by Pröbster and Diehl.[24] A yet another study reported no statically significant difference in flexural strength of zirconia aged at 37°C for 1 year.[25] Shimizu *et al*. 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 h. Their investigation confirmed that there was no significant change in the flexural strength in ceramic specimens even after such a long LTD treatment.[26] An interesting finding of the present study was that the biaxial flexural strength of the CT group increased as compared to the control (C) and ML groups, however, was lesser as compared to the air particle abrasion (Si) and LTD groups. This can be explained by the fact that compressive force generated by tetragonal to monoclinic transformation overcomes the deteriorating effects of different surface treatments. Similar observation was reported by Kosmac *et al*. and Guazzato *et al*. in their studies.[12,27] The m values observed in various studies have shown varying results from the normal values quoted for dental ceramics to values which were found to be considerably higher.[3,14,28-30] Few groups

Table 4: Tukey's honest significant difference test results

Variable (I)	Variable (J)	Mean difference (I-J)	Mean SE	*P*	95% CI	
					Lower bound	Upper bound
Control (C)	Air particle abrasion (Si)	−127.200*	6.914	0.000*	−146.85	−107.55
	ML	2.900	6.914	0.993	−16.75	22.55
	LTD	−38.300*	6.914	0.000*	−57.95	−18.65
	CT	−27.700	6.914	0.052	−47.35	−8.05

*The mean difference is significant at $P<0.05$ level. SE: Standard error, CI: Confidence interval, ML: Mechanical loading, LTD: Low-temperature loading, CT: Cumulative treatment

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

ML: Mechanical loading, LTD: Low-temperature loading, CT: Cumulative treatment

demonstrated less Weibull modulus as compared to the control group signifying that surface treatment might have affected the reliability of the clinical performance of ceramics. However, larger Weibull values represent that there are fewer critical flaws and indicate a smaller error in the judgment of clinical strength of the said ceramics.[14] The characteristic physical and mechanical properties of zirconia are attributed to its tetragonal to monoclinic phase conversions. The observations of the present study were in agreement with the findings of the previous studies where the control (C) group consisted of 100% tetragonal zirconia.[3,12,31] Y-TPZ zirconia remains stable in tetragonal state between 1145°C to below room temperature. Different surface treatments lead to phase transformations imparting characteristic physical and mechanical properties to the said materials.[3,12,14,32,33] This could be explained by the fact that when they are exposed to stress, change in crystal cell structure occurs and this results in phase transformation including tetragonal to monoclinic phase transformation.[34,35] In the present study, the greatest amount of monoclinic phase was detected following LTD treatment. Similar results were found in previous studies conducted by Kosmac *et al*.[3] and de Kler *et al*.[36] Furthermore, few studies have shown a complete absence of monoclinic content in the control (C) group.

Conclusions

The conclusions from the present study were as follows:

1. The highest biaxial flexural strength was observed in air particle abrasion (Si) group followed by the LTD group, CT group and least with the ML group
2. A 100% tetragonal zirconia was observed in the control group while the greatest amount of monoclinic percentage was observed after LTD treatment.

Limitations of study

One of the major shortcomings of the present study was that it did not mimic clinical conditions exactly which might produce different results due to the presence of saliva and pH changes wherein the restoration is actually under a set of biological conditions of moisture contamination and pH balance changes inside the oral cavity.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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