

Effects of Pretreatment, Specimen Thickness, and Artificial Aging on Biaxial Flexural Strength of Two Types of Y-TZP Ceramics

A Sundh • W Kou • G Sjögren

Clinical Relevance

Surface treatment of Y-TZP-based ceramics should be carried out with care to avoid unexpected negative effects on the material.

SUMMARY

Objectives: To evaluate the effects of surface treatment, specimen thickness, and aging on the biaxial flexural strength (BFS) of two types of yttria-stabilized, tetragonal zirconia polycrystal (Y-TZP) ceramics.

Methods and Materials: Disc-shaped specimens, 0.4 and 1.3 mm thick, made from hot isostatic pressed (Denzir) and non-hot isostatic pressed (ZirPlus) Y-TZP, were sandblasted, heat treated, and autoclaved. The surface topography was assessed in accordance with European Standard 623-624:2004 and the BFS tests in accordance with International Organi-

zation for Standardization Standard 6872:2008. For statistical analyses, one-way Shapiro-Wilk test, analysis of variance (*post hoc*: least significant differences), Mann-Whitney U-test, and Pearson correlation tests ($p < 0.05$) were used.

Results: As delivered, the BFS of the 0.4-mm ZirPlus was >1.3 -mm ZirPlus ($p < 0.01$), and the BFS of the 0.4-mm Denzir was >1.3 -mm Denzir ($p < 0.001$). Sandblasting with 0.2 MPa reduced the BFS of the ZirPlus and Denzir discs ($p < 0.01$), whereas sandblasting with 0.6 MPa increased the BFS of the 0.4-mm Denzir ($p < 0.001$) and reduced the BFS of the 0.4-mm ZirPlus ($p < 0.05$). Heat treatment significantly reduced the BFS of all the groups except for the 0.6 MPa sandblasted 0.4-mm ZirPlus. Autoclaving reduced the BFS of the as-delivered ZirPlus and Denzir specimens ($p < 0.001$), whereas autoclaving the 0.6 MPa sandblasted and heat-treated specimens had no effect ($p > 0.05$) on the BFS. The 0.6 MPa sandblasted, heat-treated, and autoclaved 0.4-mm Denzir exhibited higher BFS than the 0.6 MPa sandblasted, heat-treated, and autoclaved 0.4-mm ZirPlus ($p < 0.05$).

Anders Sundh, PhD, DDS, Praktikertjänst AB, Skellefteå, Sweden

Wen Kou, PhD, DDS, Dental Materials Science, Faculty of Medicine, Umeå University, Umeå, Sweden

*Göran Sjögren, PhD, DDS, professor, Dental Materials Science, Faculty of Medicine, Umeå University, Umeå, Sweden

*Corresponding author: Dental Materials Science, Faculty of Medicine, Umeå University, Umeå SE-901 87, Sweden; E-mail: Goran.Sjogren@umu.se

DOI: 10.2341/18-071-L

Conclusions: Thickness and surface treatment of Y-TZP-based ceramics should be considered since those factors could influence the BFS of the material.

INTRODUCTION

Zirconia was introduced into dentistry at the beginning of the 1990s, and various types of zirconia systems are currently available as biomaterials.¹⁻³ The zirconia (ZrO_2) ceramic is polymorphic and exists in three different phases—monoclinic, tetragonal, and cubic⁴—and compared with alumina and other dental ceramics has higher flexural strength and fracture resistance, making the ZrO_2 -ceramic of particular interest as a material for dental cores/substructures.¹⁻³ In addition, ZrO_2 ceramics are opaque and have a relatively small particle size, which has been said to allow the possibility of minimally invasive restorations and the masking of discolored underlying tooth structure.⁵

Today, zirconia restorations for dental application are manufactured mainly of yttria tetragonal zirconia polycrystals (Y-TZP) using computer-aided design/computer-aided manufacturing (CAD/CAM) techniques,^{1,3} and the restorations can be processed with hard or soft machining.⁵⁻⁷ Hard machining uses sintered Y-TZP blocks subjected to the process of hot isostatic pressing (HIPing), which has been said to effectively reduce porosity and possibly improve the mechanical properties of the material.⁸ Soft machining uses mainly Y-TZP presintered prefabricated blocks; an enlarged core/substructure for a crown or fixed partial denture (FPD) is machined from the presintered block and subsequently sintered to the desired dimensions before veneering.^{6,7} The recommended thickness of conventional cores/frameworks made of Y-TZP ceramics has been said to have a usual range of between 0.3 and 0.6 mm.⁹

To improve the esthetic appearance, the Y-TZP cores/frameworks are usually layered with feldspar-based or glass ceramics. During the veneering process, the cores/frameworks are subjected to heat treatment up to around 900°C-1000°C, and it has been shown that heat treatment, like veneering, significantly affects the strength of zirconia.¹⁰⁻¹²

Various other mechanical processes, such as grinding, milling, and sandblasting, could affect the properties of zirconia dental restorations.¹⁰⁻¹⁵ However, conflicting results concerning the effects of grinding/machining on the strength of zirconia ceramics have been reported.¹³⁻¹⁵ For example, some studies have shown that grinding/machining can

reduce the strength of zirconia,^{13,15} whereas other studies show that grinding/machining increases the strength.^{14,16} Moreover, surface treatments such as sandblasting have been shown to increase the biaxial flexural strength (BFS) of zirconia.^{13,15} In addition, an earlier study demonstrated that reduced thickness of HIPed Y-TZP copies could affect the fracture resistance of veneered stylized all-ceramic crowns.¹⁷

Since the thickness of the material in dental cores/substructures is important and various surface and heat treatments are usually inevitable steps in the manufacturing and veneering processes of dental ceramic restorations, the aim of the present study was to examine the effect on BFS of HIPed and unHIPed Y-TZP ceramics of 1) specimen thickness, 2) heat treatment, 3) sandblasting using various pressure, and 4) artificial aging.

METHODS AND MATERIALS

The materials studied were unHIPed Y-TZP (Zir-Plus, Cad.esthetics AB, Skellefteå, Sweden) and HIPed Y-TZP (Denzir, Cad.esthetics AB) ceramics (Figure 1a,b). The composition of the two types of ceramics was identical.

Preparation of Specimens

Two hundred disc-shaped (13-mm diameter) specimens were CAD/CAM manufactured from prefabricated blanks using the Cad.esthetics software system (Cad.esthetics AB). The ZirPlus and Denzir specimens were made in two different thicknesses: 0.4 and 1.3 mm. Means \pm SD of the thickness of the Denzir discs ($n=20$) and the ZirPlus discs ($n=20$) were 1.3 ± 0.02 mm. Corresponding figures for the ZirPlus ($n=80$) and Denzir discs ($n=80$) were 0.4 ± 0.03 and 0.4 ± 0.05 mm, respectively. Figure 1a,b summarizes the various groups of the ZirPlus and Denzir specimens studied. The reason why these thicknesses (1.3 and 0.4 mm) were chosen was because the International Organization for Standardization (ISO) Standard 6872:2008¹⁸ states that the thickness of the specimen should be 1.2 ± 0.2 mm, whereas the thickness of dental zirconia cores often is between 0.3 and 0.6 mm.^{9,17}

Ten specimens of each type and thickness of the Y-TZP ceramic were studied as delivered, that is, directly after machining. In addition, 10 specimens of each thickness and type of Y-TZP were heat treated before testing in a similar way to veneering with a feldspar-based porcelain (VITA VM9, VITA Zahnfabrik, Bad Säckingen, Germany).¹⁹ To study the effect of surface treatment and aging, before

Fig. 1a

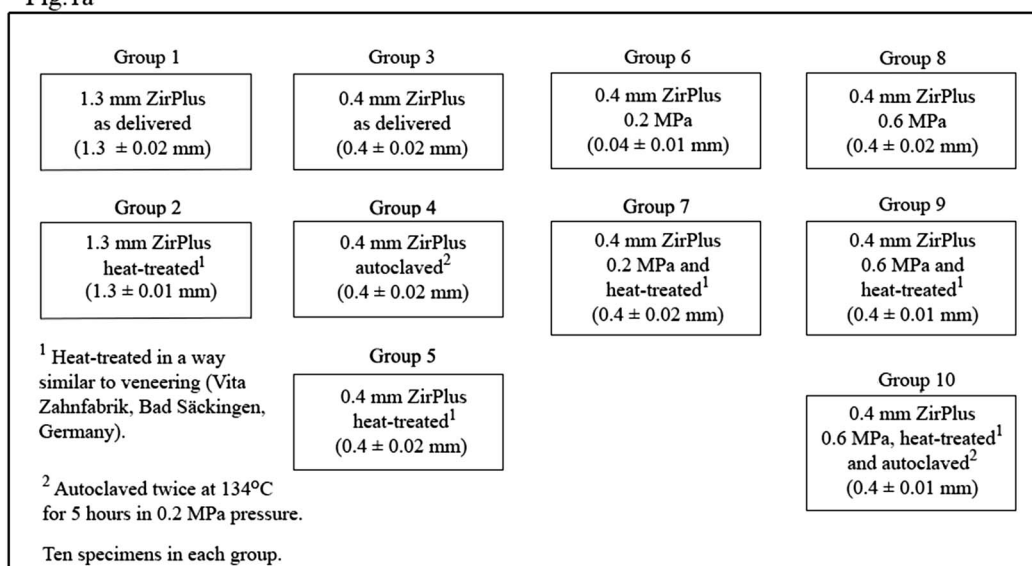


Fig. 1b

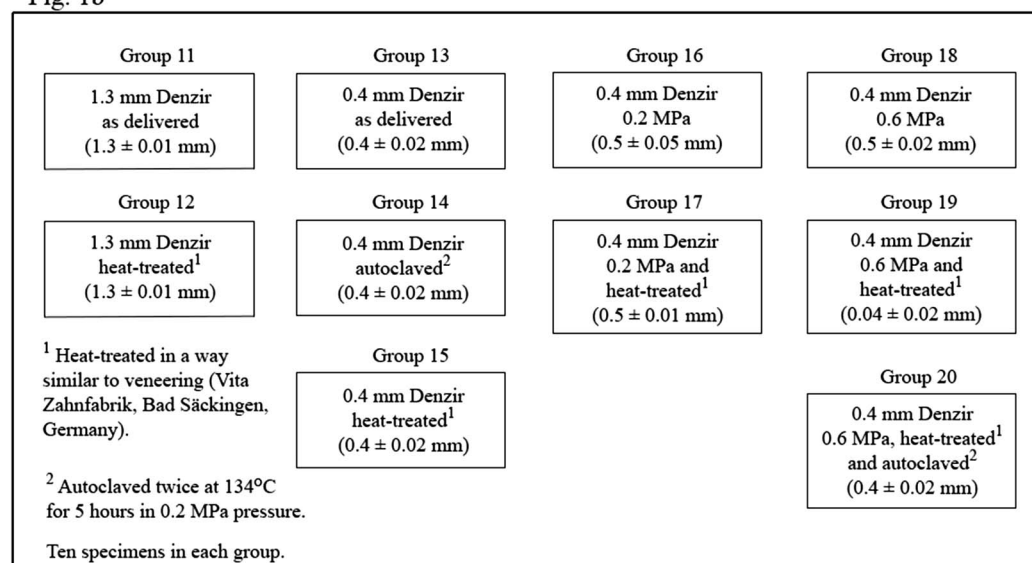


Figure 1a,b. Flowchart of the ZirPlus and Denzir test samples. Means \pm SD of the thickness of each group of the ZirPlus and Denzir discs studied are shown in parentheses. Ten specimens in each group.

testing, the tensile surface of 50 discs of the 0.4-mm ZirPlus and 50 discs of the 0.4-mm Denzir discs were sandblasted (Basic Quattro, Renfert GmbH, Hitzingen, Germany) for 90 seconds with $110 \mu\text{m Al}_2\text{O}_3$ at a distance of ~ 2 mm between the surface of the disc and the nozzle (Basic Quattro). Of these 100 discs, the tensile surfaces of 20 of the 0.4-mm ZirPlus discs and 20 of the 0.4-mm Denzir discs were sandblasted with 0.2 MPa, and the tensile surfaces of 30 of the 0.4-mm ZirPlus discs and 30 of the 0.4-mm Denzir discs were sandblasted with 0.6 MPa (Figure 1a,b). Thereafter, 10 discs of each type were subjected to

heat treatment similar to veneering (VITA VM9),¹⁹ and 10 discs of each type of the as-delivered specimens were autoclaved (Kebo Lab AB, Stockholm, Sweden) twice for five hours at 134°C in 0.2 MPa pressure.²⁰ In addition, 10 discs of each type of the sandblasted 0.4-mm discs were heat treated in a way similar to veneering (VITA VM9)¹⁹ and autoclaved twice for five hours at 134°C in 0.2 MPa pressure before testing (Figure 1a,b).²⁰ Between the two autoclave treatments, the autoclaved specimens were stored at room temperature for 16 hours.

Determining the Surface Roughness

Using a measuring profilometer (Taylor/Hobson Precision Form Taylor Surf 50, Taylor Hobson, Warrenville, IL, USA), the arithmetical mean deviation of the assessed surface roughness (Ra), the maximum profile valley depth (Rv), and the maximum height of the roughness (Rz) of the surfaces intended to be subject to tensile stress during the testing were measured; the cutoff length was 0.8 mm, evaluation length 4 mm, stylus speed 0.5 mm/s, and stylus tip radius 2 μ m, in accordance with European Standard 623-624:2004.²¹ Before testing, the specimens were cleaned ultrasonically (Branson B221, Branson Ultrasonic Co, Danbury, CT, USA) for five minutes in tap water, rinsed in distilled water, and air-dried. Five parallel scans were made at randomly selected locations in the center of the specimen on each measured surface. All measurements were recorded by one operator.

Test of Biaxial Flexural Strength

A universal testing machine (Tinius Olsen H10K-T, Horsham, PA, USA) was used for the BFS test, and the test setup was in accordance with ISO 6872:2008.¹⁸ The support area for the specimens comprised three symmetrically placed steel balls, 3.2 mm in diameter, positioned 120° apart creating a circle with a diameter of 10 mm. The disc-shaped specimens were placed on the supporting balls, and the load was applied at the center of the specimen with a flat punch, 1.6 mm in diameter, at a crosshead speed of 1 mm/min. The sandblasted surface of the specimens was placed in tension; that is, the sandblasted specimens were placed in the sample holder with the sandblasted surface face-down bearing on the three stainless-steel supporting balls.

The load in newtons (N) required to fracture the specimens was automatically recorded, and the BFS was calculated using the following equations:

$$S = -0.2387 \frac{P(X - Y)}{d^2} \quad (1)$$

where S is the maximum tensile stress (MPa) and P is the load at fracture (N) and

$$X = (1 + \nu)1n(r_2/r_3)^2 + [(1 - \nu)/2](r_2/r_3)^2 \quad (2)$$

$$Y = (1 + \nu)[1 + 1n(r_1/r_3)^2] + (1 - \nu)(r_1/r_3)^2 \quad (3)$$

where ν is the Poisson ratio, r_1 is the radius of the support circle, r_2 is the radius of the loaded area

(mm), r_3 is the radius of the specimen (mm), and d is the specimen thickness (mm).

In the current study, the Poisson ratio (ν) = 0.25, r_1 = 5 mm, r_2 = 0.8 mm, and r_3 = 6.5 mm, was used. The thickness of the fractured surface of each specimen was measured using a measuring microscope (Leitz UWM-Dig-S, Ernst Leitz GmbH, Wetzlar, Germany) at 20 \times magnification at three selected measuring points: two points in the outer borders and one point in the center of the fractured discs. For each specimen, the mean value of the three measurements for each specimen was then used as the value for d in equation 1.

Statistical Analysis

The Shapiro-Wilk test was used to test normal distribution of the data. The BFS data were statistically analyzed using one-way analysis of variance supplemented with least significant differences *post hoc* tests ($p < 0.05$). As the data of the surface roughness did not meet the normality requirement the Mann-Whitney U-test, a nonparametric test was used ($p < 0.05$) to statistically analyze the Ra , Rz , and Rv values. The Pearson correlation test ($p < 0.05$) was used to correlate the determined BFS with the surface roughness, that is, the determined Ra , Rz , and Rv values for each type of group. All data were analyzed using SPSS version 24 statistical software (Statistical Package for Social Science, SPSS Inc, Chicago, IL, USA).

RESULTS

Figure 2 presents the BFS of all the specimens studied, and Tables 1 and 2 present the statistical analyses of the BFS. Tables 3 through 5 show the arithmetical mean deviation and statistical analyses of the assessed roughness (Ra), the maximum valley depth (Rv), and the maximum height of the roughness (Rz).

Effects of Specimen Thickness, Heat Treatment, Sandblasting, and Autoclaving on the BFS

As delivered, the BFS of the 0.4-mm ZirPlus discs was significantly superior to the 1.3-mm ZirPlus discs ($p < 0.01$), and the BFS of the 0.4-mm Denzir was significantly superior ($p < 0.001$) to the 1.3-mm Denzir (Figure 2; Table 1). Sandblasting with 0.2 MPa reduced the BFS of the 0.4-mm ZirPlus and Denzir as-delivered discs ($p = 0.000$), whereas sandblasting with 0.6 MPa increased the BFS of the 0.4-mm Denzir ($p < 0.001$) and reduced the BFS of the

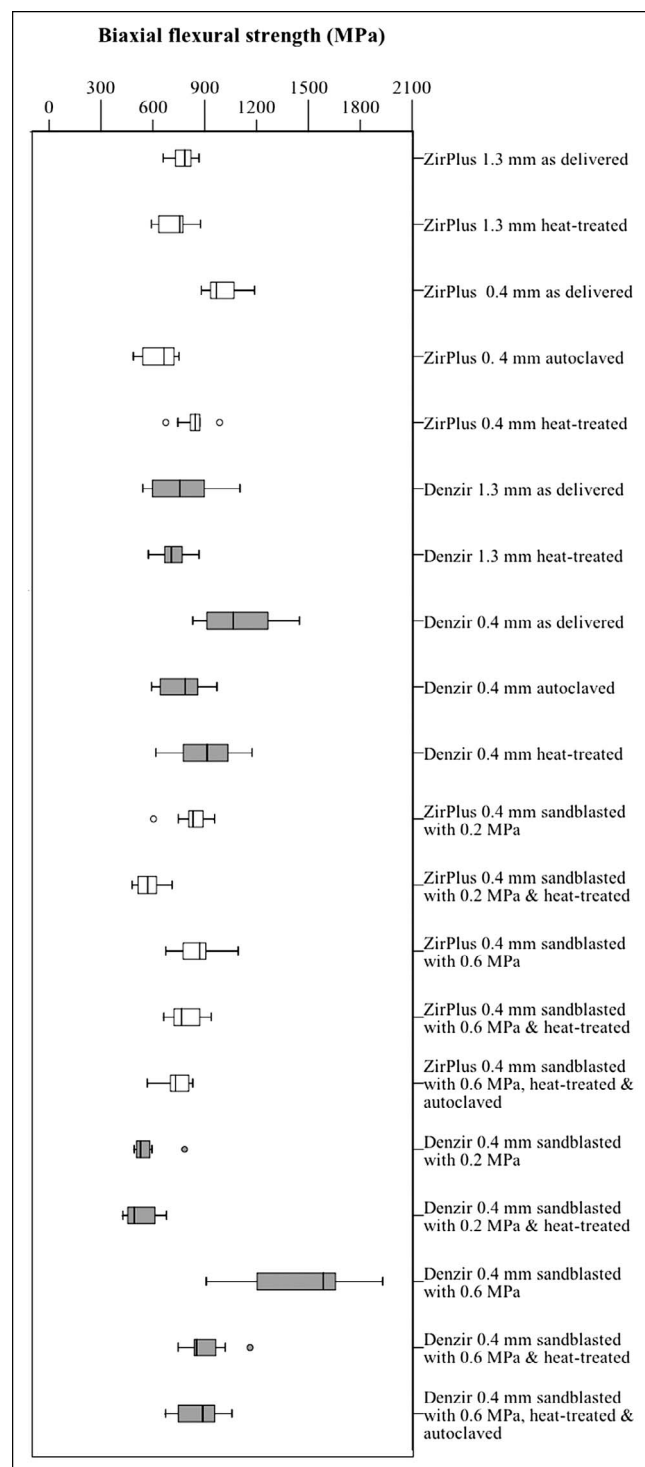


Figure 2. Box plot of the biaxial flexural strength (MPa) of all the various groups studied. Ten specimens in each test group. Data are presented as medians and first and third quartiles. The median is presented by a horizontal line within the box. The maximum and minimum values are illustrated via the upper and lower strokes. All the sandblasted surfaces of the specimens were placed in tension. White boxes: ZirPlus specimens. Gray boxes: Denzir specimens.

0.4-mm ZirPlus ($p < 0.05$). Heat treatment significantly reduced the BFS of all the groups except for the 0.6 MPa sandblasted 0.4-mm ZirPlus. Autoclaving reduced the BFS of the as-delivered ZirPlus and Denzir specimens ($p = 0.000$), whereas autoclaving the 0.6 MPa sandblasted and heat-treated 0.4-mm specimens had no effect ($p > 0.05$) on the BFS (Figure 2; Table 2). The 0.6 MPa sandblasted, heat-treated, and autoclaved 0.4-mm Denzir exhibited significantly ($p < 0.05$) higher BFS than the 0.6 MPa sandblasted, heat-treated, and autoclaved 0.4-mm ZirPlus (Figure 2; Table 2).

Surface Roughness and the Pearson Correlation Test

Of all the groups studied, the 0.6 MPa sandblasted ZirPlus discs exhibited the highest R_a , R_v , and R_z values (Tables 3-5). For the ZirPlus specimens, the Pearson correlation test revealed no statistically significant ($p > 0.05$) correlations except between R_v and BFS of one of the 0.6 MPa sandblasted, heat-treated, and autoclaved 0.4-mm ZirPlus discs ($r = 0.658$, $p = 0.039$). For the Denzir specimens, the Pearson correlation test showed no statistically significant ($p > 0.05$) correlations except for one specimen: one of the heat-treated 0.4-mm Denzir discs exhibited R_a and BFS ($r = -0.721$, $p = 0.019$), R_v and BFS ($r = -0.776$, $p = 0.008$), and R_z and BFS ($r = -0.731$, $p = 0.016$).

DISCUSSION

Effects of Specimen Thickness

Today, minimally invasive restorations with high-level-esthetic appearance have become available for dental restorations, mainly because of the introduction of oxide-based ceramics,^{1-3,10,17} and it has been stated that the small particle size of zirconia makes it possible to produce thin ceramic dental restorations.⁵ However, the quality of zirconia can be affected, for example, by the grain size, type of stabilizing oxide, heat treatment, manufacturing process, and composition,^{8,12,17,22-24} and the introduction of zirconia into dentistry has led to a number of questions about the proper handling of zirconia for dental applications. For example, in an earlier study,¹⁷ stylized, veneered ceramic crowns with cores made of HIPed Y-TZP, 0.2 mm thick and placed on a slice-formed preparation, exhibited superior fracture strength compared to the values reported in a similar study of HIPed Y-TZP ceramic crowns with 0.5-mm cores placed on a circumferential chamfer.¹⁰ In these previous studies,^{10,17} it was proposed that the monoclinic phase/tetragonal phase

Table 1: Summary of the Statistical Analysis (Analysis of Variance Supplemented With Least Significant Differences Post Hoc Test) of the Biaxial Flexural Strength of the As-Delivered and Heat-Treated ZirPlus and Denzir Specimens (10 Specimens in Each Group)^a

Specimen	A	B	C	D	E	F	G	H	I	J
A. ZirPlus, 1.3 mm, as delivered										
B. ZirPlus, 1.3 mm, heat treated	0.407									
C. ZirPlus, 0.4 mm, as delivered	0.000	0.000								
D. ZirPlus, 0.4 mm, autoclaved	0.029	0.174	0.000							
E. ZirPlus, 0.4 mm, heat treated	0.204	0.037	0.026	0.001						
F. Denzir, 1.3 mm, as delivered	0.840	0.530	0.000	0.048	0.141					
G. Denzir, 1.3 mm, heat treated	0.399	0.988	0.000	0.178	0.035	0.520				
H. Denzir, 0.4 mm, as delivered	0.000	0.000	0.090	0.000	0.000	0.000	0.000			
I. Denzir, 0.4 mm, autoclaved	0.877	0.500	0.000	0.043	0.155	0.963	0.490	0.000		
J. Denzir, 0.4 mm, heat treated	0.053	0.006	0.118	0.000	0.503	0.033	0.006	0.001	0.037	

^a Statistical differences ($p < 0.05$). The values in the table refer to the p-values obtained.

distribution in the Y-TZP core could have influenced the outcome.

Similarly, in the present study the BFS of the as-delivered 0.4-mm Denzir discs, that is, before a heat treatment similar to veneering, was significantly superior to the as-delivered 1.3-mm Denzir discs, and the BFS of the as-delivered 0.4-mm ZirPlus discs was superior to that of the as-delivered 1.3-mm ZirPlus discs (Figure 2; Table 1). That is, thinner specimens exhibited significantly ($p=0.000$) higher BFS than thicker specimens of a similar type of material. One possible explanation for thinner discs made of Y-TZP ceramics resulting in higher BFS could be the phase transformation $t \rightarrow m$ created on the surface because of the machining process. A monoclinic layer could be detected on ground/machined surfaces of Y-TZP ceramic,²⁵ and a proportionally thicker monoclinic layer may be created on thinner specimens than on thicker

specimens. Since the monoclinic layer has ~3%-4% volume expansion compared to the tetragonal phase, the compressive layer formed at the surface by this layer could have resulted in the thinner specimens having a higher flexural strength than the thicker specimens.¹³ It has been reported in a study by Ramos and others²⁵ that higher monoclinic content could be detected on ground zirconia surfaces compared to nonground surfaces and that subsequent heat treatment produced reversed transformation.²⁶ On the other hand, in a previous study of magnesia-stabilized zirconia (Mg-PSZ), the various specimen thicknesses, that is, 0.4-mm and 1.3-mm discs, did not influence the BFS.² However, it has been stated that phase transformation toughening is less in Mg-PSZ than in Y-TZP,^{5,27,28} but it was also seen in the previous study of Mg-PSZ² that sandblasting and heat treatment could affect the strength of Mg-PSZ specimens. Another reason that the

Table 2: Summary of the Statistical Analysis (Analysis of Variance Supplemented With Least Significant Differences Post Hoc Test) of the Biaxial Flexural Strength of the 0.2 and 0.6 MPa Sandblasted and Autoclaved ZirPlus and Denzir Specimens (10 Specimens in Each Group)^a

Specimen	K	L	M	N	O	P	Q	R	S	T
K. ZirPlus, 0.4 mm, 0.2 MPa										
L. ZirPlus, 0.4 mm, 0.2 MPa, heat treated	0.000									
M. ZirPlus, 0.4 mm, 0.6 MPa	0.575	0.000								
N. ZirPlus, 0.4 mm, 0.6 MPa, heat treated	0.521	0.001	0.229							
O. ZirPlus, 0.4 mm, 0.6 MPa, heat treated and autoclaved	0.125	0.013	0.037	0.370						
P. Denzir, 0.4 mm, 0.2 MPa	0.000	0.771	0.000	0.000	0.006					
Q. Denzir, 0.4 mm, 0.2 MPa, heat treated	0.000	0.393	0.000	0.000	0.001	0.573				
R. Denzir, 0.4 mm, 0.6 MPa	0.000	0.000	0.000	0.000	0.000	0.000	0.000			
S. Denzir, 0.4 mm, 0.6 MPa, heat treated	0.289	0.000	0.617	0.090	0.010	0.000	0.000	0.000		
T. Denzir, 0.4 mm, 0.6 MPa, heat treated and autoclaved	0.544	0.000	0.963	0.212	0.033	0.000	0.000	0.000	0.650	

^a Statistical differences ($p < 0.05$). The values in the table refer to the p-values obtained.

Table 3: Arithmetic Mean Deviation of the Assessed Profile (Ra) of the ZirPlus and Denzir Specimens (μm)^a

Specimen	ZirPlus (Mean \pm SD)	Denzir (Mean \pm SD)
1.32 mm, as delivered	0.3 \pm 0.1 A	0.3 \pm 0.1 A, B, E
1.32 mm, heat treated	0.3 \pm 0.2 A, B	0.2 \pm 0.1 C, H
0.4 mm, as delivered	0.2 \pm 0.1 C	0.2 \pm 0.1 C, K
0.4 mm, autoclaved	0.4 \pm 0.1 D	0.4 \pm 0.2 D
0.4 mm, heat treated	0.3 \pm 0.2 A, B, C, E	0.2 \pm 0.1 C, E, H, K
0.4 mm, 0.2 MPa	0.5 \pm 0.2 F	0.6 \pm 0.2 I
0.4 mm, 0.2 MPa, heat treated	0.5 \pm 0.1 F, G	0.6 \pm 0.1 G, I
0.4 mm, 0.6 MPa	1.3 \pm 0.2	0.9 \pm 0.2 J, L
0.4 mm, 0.6 MPa, heat treated	1.2 \pm 0.3	0.9 \pm 0.2 J
0.4 mm, 0.6 MPa, heat treated and autoclaved	1.4 \pm 0.2	0.8 \pm 0.1 L

^a Identical letters indicate absence of statistically significant difference ($p > 0.05$). No letter indicates statistically significant difference compared with all the other groups in respective table ($p < 0.05$). $n = 10$ in each group (Mann-Whitney U-tests).

specimen thickness affected the strength of the Y-TZP specimens in the present study could be that the volumes between the 0.4-mm and the 1.3-mm specimens were different. That is, when the volume of the specimens increases, it will be more likely to hit strength-limiting defects in the ceramics, which could decrease the mean strength, that is, the effect of volume scaling.²⁹ Thus, when evaluating the results of, among others, the BFS tests, the thickness of the zirconia samples should be taken into consideration.

Effects of Surface Treatment

During the manufacturing processes of dental restorations, the surfaces of copies/frameworks are subjected to mechanical treatments, such as grinding, polishing, and/or sandblasting,^{30,31} and previous studies show that the effects on the mechanical properties of such surface treatments and heat treatment of zirconia can vary.^{13,32-34}

In order to mimic surface treatments in the present study, one of the flat surfaces of the 0.4-mm disc-shaped specimens was subjected to sandblasting using two different pressures: 0.2 MPa and 0.6 MPa. Sandblasting the 0.4-mm ZirPlus and 0.4-mm Denzir discs with 0.2 MPa significantly reduced BFS compared to the as-delivered specimens, whereas when a pressure of 0.6 MPa was used, the BFS of the 0.4-mm Denzir discs was significantly superior to the as-delivered discs, while the BFS of the 0.4-mm ZirPlus discs was reduced compared to the as-delivered discs. Possible explanations could be that 0.2 MPa sandblasting mimicked polishing rather than grinding of the specimen surfaces and that 0.6 MPa sandblasting caused a rougher surface of the ZirPlus discs than of the Denzir discs (Tables 3-5). It has also been demonstrated previously that surface treatments of zirconia could produce a variety of effects.^{31,35-37} The study by Hjerpe and others³¹ shows that flexural strength could be improved by surface conditioning with airborne-particle abrasion.

Table 4: Maximum Profile Valley Depth (Rv) of the ZirPlus and Denzir Specimens (μm)^a

Specimen	ZirPlus (Mean \pm SD)	Denzir (Mean \pm SD)
1.32 mm, as delivered	1.5 \pm 0.5 A, E	1.0 \pm 0.8 C
1.32 mm, heat treated	1.4 \pm 0.5 A, B, E	0.5 \pm 0.3 I
0.4 mm, as delivered	0.9 \pm 0.4 C	0.6 \pm 0.3 I, J
0.4 mm, autoclaved	1.5 \pm 0.5 A, B, D	1.2 \pm 0.5 D, E
0.4 mm, heat treated	1.3 \pm 0.8 A, B, D, E	0.7 \pm 0.3
0.4 mm, 0.2 MPa	2.4 \pm 0.8 F, M	1.5 \pm 0.4 A, B, D, E, K
0.4 mm, 0.2 MPa, heat treated	2.6 \pm 0.7 F	1.5 \pm 0.3 A, B, D, E, K
0.4 mm, 0.6 MPa	4.5 \pm 1.1 G	2.2 \pm 0.4 L, M
0.4 mm, 0.6 MPa, heat treated	4.5 \pm 1.2 G	2.1 \pm 0.4 L
0.4 mm, 0.6 MPa, heat treated and autoclaved	5.4 \pm 0.9	2.1 \pm 0.4 L, M

^a Identical letters indicate absence of statistically significant difference ($p > 0.05$). No letter indicates statistically significant difference compared with all the other groups in respective table ($p < 0.05$). $n = 10$ in each group (Mann-Whitney U-tests).

Table 5: Maximum Height of the Profile (Rz) of the ZirPlus and Denzir Specimens (μm) ^a		
Specimen	ZirPlus (Mean±SD)	Denzir (Mean±SD)
1.32 mm, as delivered	2.5 ± 0.7 A	1.4 ± 0.4
1.32 mm, heat treated	2.5 ± 1.0 A, B	1.0 ± 0.5 F
0.4 mm, as delivered	1.8 ± 0.7	1.0 ± 0.5 F
0.4 mm, autoclaved	2.8 ± 0.9 A, B, C	2.2 ± 0.9 A, B
0.4 mm, heat treated	2.6 ± 1.5 A, B, C	1.2 ± 0.5 F
0.4 mm, 0.2 MPa	4.6 ± 1.4 D	3.1 ± 0.8 C
0.4 mm, 0.2 MPa , heat treated	4.5 ± 1.1 D	3.1 ± 0.7 C
0.4 mm, 0.6 MPa	8.6 ± 1.9 E	4.4 ± 0.9 D, G
0.4 mm, 0.6 MPa, heat treated	8.2 ± 2.1 E	4.2 ± 0.8 D, G, H
0.4 mm, 0.6 MPa, heat treated and autoclaved	9.5 ± 1.4	4.1 ± 0.7 D, H
^a Identical letters indicate absence of statistically significant difference (p>0.05). No letter indicates statistically significant difference compared with all the other groups in respective table (p<0.05). n = 10 in each group (Mann-Whitney U-tests).		

The studies by Garcia Fonseca and others³⁴ and Özcan and others³⁶ demonstrate that the size and type of particles could affect the strength of Y-TZP ceramic and reduce it; these findings provide one possible explanation for the effect of 0.6 MPa sandblasting on the ZirPlus discs. Manufacturing and adjustments involving grinding and/or polishing of zirconia-based restorations could influence the BFS of Y-TZP ceramics and should therefore be carried out carefully.

Effects of Heat Treatment and Accelerated Aging

Heat treatment did not significantly affect the BFS of the 1.3-mm specimens but significantly reduced the BFS of the 0.4-mm discs (Figure 2; Table 1). For the 0.2 MPa sandblasted discs, only the ZirPlus specimens were affected by the heat treatment, whereas heat treatment of the 0.6 MPa sandblasted discs significantly reduced the BFS of both the ZirPlus and the Denzir specimens (Figure 2; Table 2). Another interesting result was that autoclaving did not affect the BFS after heat treatment of the 0.6 MPa sandblasted 0.4-mm ZirPlus and Denzir specimens, whereas the BFS of the as-delivered 0.4-mm specimens was reduced after autoclaving (Figure 2; Tables 1 and 2). These effects on the BFS of autoclave treatment of the ZirPlus and Denzir discs in the current study were similar to the findings reported in the previous study of Mg-PSZ specimens.² In that study, it was shown that autoclaving of the as-delivered Mg-PSZ specimens reduced their strength, whereas it had no influence on the BFS of heat-treated 0.6 MPa sandblasted specimens.

The fact that the heat-treated discs were not affected by subsequent autoclaving is of interest since accelerated aging of ceramics by autoclaving at

134°C for five hours has been recommended in ISO 13356; 2015³⁸ as a method for studying aging of, for example, ceramic femoral heads. In the current study, the specimens were autoclaved at 134°C and 0.2 MPa for 10 hours, which, according to Chevalier and others,³⁹ corresponds to ~36-48 years *in vivo*. In the previous study by Lucas and others²⁴ of two brands of Y-TZP, autoclaving for five hours and 0.2 MPa did not influence the modulus of elasticity. It has also been reported in previous studies of zirconia^{5,24} that the grain size could influence the t → m transformation²³ and that grains below a certain size seem not to transform t → m.⁵ However, it should be noted that in a meta-analysis by Pereira and others³⁹ of low-temperature degradation of Y-TZP ceramic, it was stated that autoclave aging could reduce the mechanical properties of Y-TZP and that aging time, pressure, and temperature affect the outcome. Another meta-analysis⁴⁰ reported that airborne-particle abrasion could both increase and reduce the strength and affect the phase transformation in Y-TZP and that after artificial aging in an autoclave for more than 12 hours, there was less monoclinic content in the sandblasted specimens than in the control. In the present study, both autoclaving and heat treatment could reduce the BFS, and it is of great interest to examine how these methods affect the phase transformation of the studied Y-TZP materials, and further studies are needed to clarify the transformation process.

Surface Roughness and the Pearson Correlation Test

The Ra, Rv, and Rz values of the 0.4-mm ZirPlus and 0.4-mm Denzir specimens in the current study were significantly increased after autoclaving compared to the as-delivered discs (Tables 3 through 5). This is in

agreement with the results in a previous study by Lucas and others²⁴ addressing the artificial aging of zirconia in autoclaving where a slight increase in surface roughness was seen after aging in an autoclave for five hours. In spite of this, the Pearson correlation test in the present study showed no correlation ($p < 0.05$) between the BFS and the surface roughness, except for the heat-treated 0.4-mm Denzir discs and the heat-treated and autoclaved 0.4-mm ZirPlus discs sandblasted with 0.6 MPa. Similar findings were reported in the study by Ramos and others,²⁶ in which it was stated that no correlation was seen between surface roughness and the BFS of Y-TZP ceramic. On the other hand, in the study by Hjerpe and others,³¹ a statistically significant correlation was seen between surface roughness and the strength values in a three-point bend test of zirconia, indicating that the surface roughness could influence the fracture behavior of dental zirconia.

CONCLUSIONS

Based on the findings in the present study, several factors could significantly influence the BFS of the Y-TZP-based ceramics studied. In particular, the thickness of Y-TZP-based ceramics seems to have a major impact on the BFS of the material. In addition, surface treatments, such as sandblasting and heat treatment, have an impact on the BFS in the Y-TZP ceramics tested. Since heat treatment and sandblasting are methods that are commonly used for manufacturing this type of dental ceramic restoration, the surface treatment of Y-TZP ceramics should be carried out with care to avoid unexpected negative effects on the material.

Acknowledgement

The authors would like to thank Cad.esthetics AB, Skellefteå, Sweden, for kindly donating the specimens.

Conflict of Interest

Dr Anders Sundh has previously been employed (part-time) as manager of research and development at Cad.esthetics AB, Skellefteå, Sweden.

(Accepted 30 July 2018)

REFERENCES

1. Häff A, Löf H, Gunne J, & Sjögren G (2014) A retrospective evaluation of zirconia-fixed partial dentures in general practices: An up to 13-year study *Dental Materials* **31**(2) 162-170.
2. André M, Kou W, Sjögren G, & Sundh A (2016) Effects of pretreatments and hydrothermal aging on biaxial flexural strength of lithium di-silicate and Mg-PSZ ceramics *Journal of Dentistry* **55** 25-32.
3. Ioannidis A & Bindl B (2016) Clinical prospective evaluation of zirconia-based three-unit posterior fixed dental prostheses: Up-to ten-year results *Journal of Dentistry* **47** 80-85.
4. Kisi EH & Howard CJ (1998) Crystal structures of zirconia phases and their inter-relation *Key Engineering Materials* **153-154** 1-36.
5. Denry I & Kelly JR (2008) State of the art of zirconia for dental applications *Dental Materials* **24**(3) 299-307.
6. Filser F, Kocher P, Weibel F, Luthy H, Scharer P, & Gauckler LJ (2001) Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM) *International Journal of Computerized Dentistry* **4**(2) 89-106.
7. Al-Amleh B, Lyons K, & Swain M. Clinical trials in zirconia: A systematic review (2010) *Journal of Oral Rehabilitation* **37**(8) 641-652.
8. Tsukuma K & Shimada M (1985) Hot isostatic pressing of Y_2O_3 -partially stabilized zirconia *American Ceramic Society Bulletin* **2**(64) 310-313.
9. Parker RM (2007) Use of zirconia in restorative dentistry *Dentistry Today* **26**(3) 114-119.
10. Sundh A & Sjögren G (2004) A comparison of fracture strength of yttrium-oxide-partially-stabilized zirconia ceramic crowns with varying core thickness, shapes and veneer ceramics *Journal of Oral Rehabilitation* **31**(7) 682-688.
11. Sundh A, Molin M, & Sjögren G (2005) Fracture resistance of yttrium oxide partially-stabilized zirconia all-ceramic bridges after veneering and mechanical fatigue testing *Dental Materials* **21**(5) 476-482.
12. Sundh A & Sjögren G (2006) Fracture resistance of all-ceramic zirconia bridges with differing phase stabilizers and quality of sintering *Dental Materials* **22**(8) 778-784.
13. Kosmač T, Oblak C, Jevnikar P, Funduk N, & Marion L (1999) The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic *Dental Materials* **15** (6) 426-433.
14. Guazzato M, Quach L, Albakry M, & Swain MV (2005) Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic *Journal of Dentistry* **33**(1) 9-18.
15. Karakoca S & Yilmaz H (2009) Influence of surface treatments on surface roughness, phase transformation, and biaxial flexural strength of Y-TZP ceramics. *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **91**(2) 930-937.
16. Qeblawi DM, Muñoz CA, Brewer JD, & Monaco EA Jr (2010) The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. *Journal of Prosthetic Dentistry* **103**(4) 210-220.
17. Åkesson J, Sundh A, & Sjögren G (2009) Fracture resistance of all-ceramic crowns placed on a preparation with a slice-formed finishing line *Journal of Oral Rehabilitation* **36**(7) 516-523.

18. ISO-Standards (2008) ISO 6872 Dentistry, ceramic materials *Geneve: International Organization for Standardization*.
19. VITA VM9 Working Instructions (2013) *Bad Säckingen, Germany: VITA Zahnfabrik*.
20. ISO-Standards (2015) ISO Standard 13356 Implants for surgery – Ceramic materials based on yttria stabilized tetragonal zirconia (Y-TZP). *Geneve: International Organization for Standardization*.
21. European Standard (2004) EN 623-624 Advanced technical ceramics – Monolithic ceramics – General and textural properties. Part 4: Determination of surface roughness. *Brussels: European Committee for Standardization*.
22. Piconi C & Maccauro G (1999) Zirconia as a ceramic biomaterial *Biomaterials* **20**(1) 1-25.
23. Grant KL, Rawlings RD, & Sweeney R (2001) Effect of HIPping, stress and surface finish on the environmental degradation of Y-TZP ceramics *Journal of Materials Science: Materials in Medicine* **12**(6) 557-564.
24. Lucas TJ, Lawson NC, Janowski GM, & Burgess JO (2015) Effect of grain size on the monoclinic transformation, hardness, roughness, and modulus of aged partially stabilized zirconia *Dental Materials* **31**(12) 1487-1492.
25. Chevalier J, Gremillard L, Virkar AV, & Clarke DR (2009) The tetragonal-monoclinic transformation in zirconia: Lessons learned and future trends *Journal of the American Ceramic Society* **92**(9) 1901-1920.
26. Ramos GF, Pereira GK, Amaral M, Valandro LF, & Bottino MA (2016) Effect of grinding and heat treatment on the mechanical behavior of zirconia ceramic *Brazilian Oral Research* 30, doi:10.1590/1807-3107BOR-2016.vol30.0012
27. Roy ME, Whiteside LA, Katerberg BJ, Steiger JA, & Nayfeh T (2007) Not all zirconia femoral heads degrade in vivo *Clinical Orthopaedics and Related Research* **465** 220-226.
28. Cales B (2000) Zirconia as a sliding material: Histologic, laboratory, and clinical data (2000) *Clinical Orthopaedics and Related Research* **379** 94-112.
29. Fleming GJP, Cao X, Romanyk DL, & Addison O (2017) Favorable residual stress induction by resin-cementation on dental porcelain *Dental Materials* **33**(11) 1258-1265.
30. Canneto JJ, Cattani-Lorente M, Durual S, Wiskott AH, & Scherrer SS (2016) Grinding damage assessment on four high-strength ceramics *Dental Materials* **32**(2) 171-182.
31. Hjerpe J, Närhi TO, Vallittu PK, & Lassila LV (2016) Surface roughness and the flexural and bend strength of zirconia after different surface treatments. *Journal of Prosthetic Dentistry* **116**(4) 577-583.
32. Mohammadi-Bassir M, Babasafari M, Rezvani MB, & Jamshidian M (2017) Effect of coarse grinding, over-glazing, and 2 polishing systems on the flexural strength, surface roughness, and phase transformation of yttrium-stabilized tetragonal zirconia *Journal of Prosthetic Dentistry* **118**(5) 658-665.
33. Inokoshi M, Zhang F, Vanmeensel K, De Munck J, Minakuchi S, Naert I, Vleugels J, & Van Meerbeek B (2017) Residual compressive surface stress increases the bending strength of dental zirconia *Dental Materials* **33**(4) e147-e154.
34. Passos SP, Linke B, Major PW, & Nychka JA (2015) The effect of air-abrasion and heat treatment on the fracture behavior of Y-TZP *Dental Materials* **3**(9) 1011-1021.
35. Garcia Fonseca R, de Oliveira Abi-Rached F, dos Santos Nunes Reis JM, Rambaldi E, & Baldissara P (2013) Effect of particle size on the flexural strength and phase transformation of an airborne-particle abraded yttria-stabilized tetragonal zirconia polycrystal ceramic *Journal of Prosthetic Dentistry* **110**(6) 510-514.
36. Özcan M, Melo RM, Souza RO, Machado JP, Felipe Valandro L, & Bottino MA (2013) Effect of air-particle abrasion protocols on the biaxial flexural strength, surface characteristics and phase transformation of zirconia after cyclic loading *Journal of the Mechanical Behavior of Biomedical Materials* **20** 19-28.
37. Bagheri H, Tabassom Hooshmand T, & Aghajani F (2015) Effect of ceramic surface treatments after machine grinding on the biaxial flexural strength of different CAD/CAM dental ceramics *Journal of Dentistry (Tehran, Iran)* **12**(9) 621-629.
38. Chevalier J, Cales B, & Drouin JM (1999) Low temperature aging of Y-TZP ceramics *Journal of the American Ceramic Society* **82**(8) 2150-2154.
39. Pereira GK, Venturini AB, Silvestri T, Dapieve KS, Montagner AF, Soares FZM, & Valandro LF (2015) Low-temperature degradation of Y-TZP ceramics: A systematic review and meta-analysis *Journal of the Mechanical Behavior of Biomedical Materials* **55** 151-163.
40. Aurélio IL, Marchionatti AM, Montagner AF, May LG, & Soares FZ (2016) Does air particle abrasion affect the flexural strength and phase transformation of Y-TZP? A systematic review and meta-analysis *Dental Materials* **32**(6) 827-845.