



Mechanical properties of zirconia after different surface treatments and repeated firings

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PURPOSE. This study investigated the influence of surface conditioning procedures and repeated firings on monoclinic content and strength of zirconia before cementation. **MATERIALS AND METHODS.** Sintered bar-shaped zirconia specimens were subjected to no surface treatment (control), air abrasion, or grinding (n=21). Their roughness was evaluated using a profilometer, and microscope analysis was performed on one specimen of each group. Then, 2 or 10 repeated firings (n=10) were executed, the monoclinic content of specimens was analyzed by X-ray diffraction, and a three-point flexural strength test was performed. Surface roughness values were compared using one-way analysis of variance (ANOVA) and Tukey honestly significant difference (HSD) tests, the monoclinic content values were tested using Kruskal-Wallis and Mann-Whitney U tests, and the flexural strength values were tested using two-way ANOVA and Tukey HSD tests ($P=.05$). Spearman's correlation test was performed to define relationships among measured parameters. **RESULTS.** Surface-treated specimens were rougher than untreated specimens and had a higher monoclinic content ($P<.005$), and the relationship between roughness and monoclinic content was significant ($P<.000$). Neither surface treatment nor firing significantly affected the flexural strength, but Weibull analysis showed that for the air-abraded samples the characteristic strength was significantly lower after the 10th firing than after the 2nd firing. **CONCLUSION.** After firing, a negligible amount of monoclinic content remained on the zirconia surfaces, and rougher surfaces had higher monoclinic contents than untreated surfaces. Multiple firings could be performed if necessary, but the fracture probability could increase after multiple firings for rougher surfaces. [J Adv Prosthodont 2014;6:462-7]

KEY WORDS: Surface Treatment; Zirconium Oxide; Ceramics; X-ray diffraction; Flexural Strength

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INTRODUCTION

Zirconia-based ceramics, such as yttria-tetragonal zirconia polycrystals (Y-TZP), are used as core materials in dental restorations.^{1,2} They consist mainly of zirconia and partially of Y_2O_3 . Zirconia can be present in different phases: cubic, tetragonal, and monoclinic.^{3,4} In Y-TZP ceramics, the tetragonal form can be stabilized at room temperature by Y_2O_3 , but external stresses can trigger a transformation from the tetragonal to monoclinic phase. This transformation is related with volume enlargement that urges compressive forces, thereby preventing further crack propagation.^{5,6} This phenomenon makes zirconia potentially superior to other dental ceramics.⁶ Furthermore, these materials have additional advantages such as high flexural strength, fracture toughness,⁷ and high biocompatibility.²

To improve its aesthetic properties, the zirconia core is often veneered with conventional feldspathic porcelain to achieve an aesthetic appearance.⁸ For this purpose, clinical and laboratory processing procedures such as sandblasting or grinding are used to achieve better adhesion between zirconia core and veneering porcelain, to improve bond between luting and prosthetic material, and during final adjustments of the restorations.⁹ However, these procedures promote the tetragonal to monoclinic phase transformation^{5,9-12} and may generate surface flaws on zirconia that can serve as stress accumulation areas.^{13,14} In addition, in clinical procedures, all-ceramic restorations are sometimes fired several times to correct the form and color of the restorations to produce a natural appearance. It was reported that heat treatments in the temperature range of 900°C–1,000°C after surface treatments of Y-TZP induce reverse phase transformation from monoclinic to tetragonal.^{11,15,16}

There have been controversial reports regarding the effects of surface^{3,5,17-19} or heat treatments^{16,20} on flexural strength of zirconia. In particular, evaluations of the effect of surface treatments have shown that the strength of the zirconia was increased^{3,19} or decreased^{5,17,18} depending on the surface damage introduced by the surface treatments. Similarly, investigations into the effects of heat treatments have reported increases²⁰ or decreases¹⁶ in strength.

These studies^{3,5,16-20} focused either on the influence of surface treatment or on the influence of heat treatment on the microstructure and strength of zirconia. However, under clinical conditions, zirconia is exposed to a series of steps consisting of surface treatments and firings. Therefore, the objective of this work was to explore the effects of surface conditioning processes and repeated firings on monoclinic content and flexural strength of zirconia before cementation. The following null hypotheses were tested: (1) different surface treatments and repeated firings do not affect the monoclinic content of zirconia and (2) different surface treatments and repeated firings do not affect flexural strength of zirconia.

MATERIALS AND METHODS

Sixty-three bars (L: 20 mm, W: 4 mm, H: 1.2 mm) were produced by cutting and sintering presintered zirconium oxide blocks (VITA In-Ceram® YZ for in Lab®; VITA Zahnfabrik, Bad Säckingen, Germany). According to the surface conditioning procedures, they were divided into three groups (n=21).

- Control: No surface conditioning was applied.
- Air abrasion: The surfaces were abraded with 110 µm Al₂O₃ particles (Korox; Bego, Bremen, Germany) from a distance of about 10 mm at a pressure of 3 bar for 10 seconds.
- Grinding: The ceramic surfaces were ground using a hand-piece (Ultimate 500K; NSK, Kanuma, Japan) at a grinding speed of 20,000 rpm for 10 seconds. During the grinding procedure, a diamond bur with a 110 µm grain size (Bredent, Senden, Germany) was used without water-spray cooling. A gentle stroking motion was employed, in order

to prevent the formation of high stress concentration areas. The burs were changed after the grinding of every five specimens. The dimensions were measured by an electronic digital micrometer (Mitutoyo Corporation, Kawasaki, Kanagawa, Japan).

A specimen from each surface treatment group was randomly selected, and its surface was examined in a scanning electron microscope (SEM; LEO 440; Zeiss, Cambridge, UK) at ×700 magnification to detect the morphological alterations of the zirconia following surface treatments.

A profilometer (Mitutoyo SurfTest 402; Mitutoyo, Minatoku, Japan) was used to determine the surface roughness (Ra in µm) of each sample. Prior to measurement, the profilometer was calibrated. Five measurements were performed at different locations near the center of the specimen, and their average was calculated to obtain the mean roughness of each sample.

Then, all specimens were subjected to heat treatments in a ceramic oven (Vita Vacumat 40T, Vita Zahnfabrik, Bad Säckingen, Germany) either 2 or 10 times to mimic the glaze firing recommended by the manufacturer (Vita, Bad Säckingen, Germany) with a starting temperature of 500°C, predrying time of 5 min, heating rate of 80°C, ending temperature of 900°C, and holding time of 1 min without a vacuum.

After heat treatment, X-ray diffraction (XRD) analysis was executed using an X-ray diffractometer (Bruker D8 Advance, Bruker, Germany) at 2θ range between 20° and 40° with a step size of 0.02° and a normalized count time of 1.8 s/step. The monoclinic phase content (%) on the surfaces was determined by using formulas (A)²¹ and (B)²² below:

$$(A) X_m = [I_m(-111) + I_m(111)] / [I_m(-111) + I_m(111) + I_t(101)]$$

$$(B) V_m = 1.311 \times X_m / 1 + (0.311 \times X_m)$$

In these formulas, X_m is the monoclinic peak intensity ratio, V_m is the monoclinic volume content (%), $I_m(-111)$ and $I_m(111)$ are the monoclinic peak intensities at $2\theta=28.2^\circ$ and 31.4° , respectively, and $I_t(101)$ is the tetragonal peak intensity at $2\theta=31.1^\circ$.²³

Finally, the flexural strengths of the bars were measured by a three-point flexural strength test apparatus (Fig. 1) according to ISO 6872:2008 at a loading rate of 1 mm/min in a universal testing machine (TSTM 02500; Elista Ltd. Şti., İstanbul, Turkey). The sample holder had a span of 15 mm between the two supports. The supports and loading piston were steel knife edges rounded to radii of 0.8 mm. The surface-treated sides were facing the loading piston, and the lower surfaces were facing the supporting device during the three-point bending test. The load was exerted at the middle of the sample. Failure load was recorded for each bar, and flexural strength data (σ in MPa) were calculated using the formula below.²⁴

$$\sigma = 3Fd / 2nb^2$$

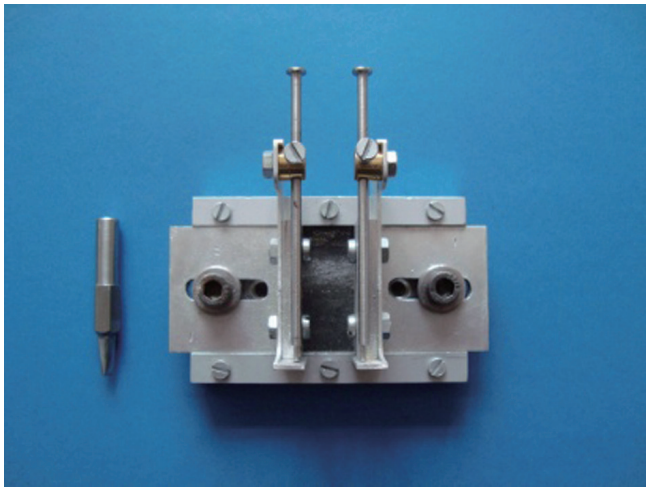


Fig. 1. Three-point flexural strength test apparatus.

In this formula, F is the fracture load (N), d is the distance between two supports (mm), w is the width of the sample (mm), and h is the height of the sample (mm).

In this study, roughness values were compared using one-way analysis of variance (ANOVA) and Tukey honestly significance difference (HSD) test, and monoclinic content values were compared by nonparametric analyses using Kruskal-Wallis and Mann-Whitney U tests. The flexural strength values were statistically tested by two-way ANOVA

and Tukey HSD tests with the surface treatments and the number of firing cycles as the independent variables. In addition, variability of the flexural strength values was tested using Weibull distribution. The Weibull modulus was calculated using the following equation.²⁵

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

In this formula, P is probability of failure, σ is flexural strength at a given P , σ_0 is characteristic strength or scale parameter at the fracture probability of 63.2%, and m is Weibull modulus which defines the structural reliability. Lower m values indicate lower reliability or vice versa.²⁵ Wald tests and 95% confidence intervals were used for the comparisons of Weibull parameters.

In addition, the relationships between the roughness and monoclinic content, between the monoclinic content and flexural strength, and between the roughness and flexural strength were assessed by Spearman's correlation analysis. In all statistical analyses a significance level of $P=.05$ was used.

RESULTS

According to SEM evaluation, control sample had a smooth surface texture with no cracks (Fig. 2A). On the other hand, morphological surface changes such as the formation of microscale retentive grooves and uniformly oriented scratches were observed in the air-abraded and ground specimens, respectively (Fig. 2B, Fig. 2C).

Table 1 lists the results of the one-way ANOVA and

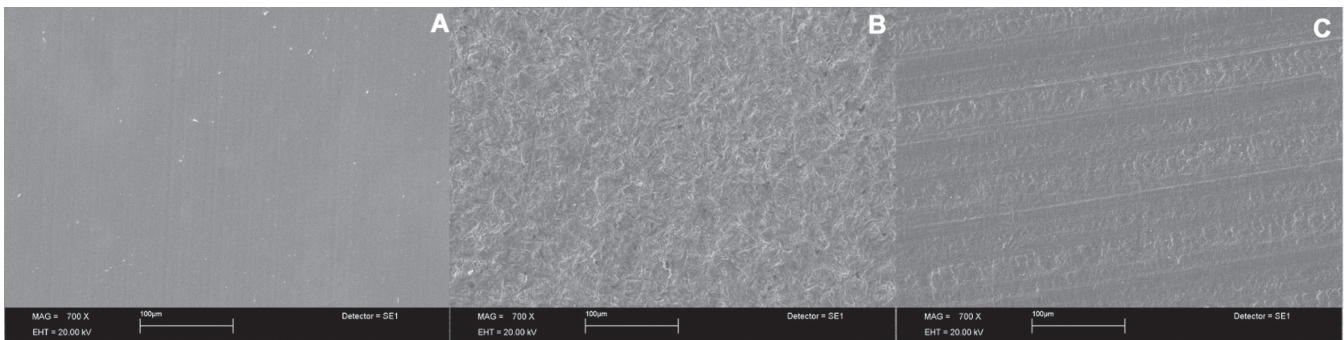


Fig. 2. SEM views of zirconia surfaces (X700). (A) Control, (B) Air-abraded, (C) Ground.

Table 1. Results of the statistical analysis of surface roughness values (Ra, µm)

Group	N	Mean	SD	Test	
Control	20	0.47 ^a	0.15	F=25.33	P<.001
Air abrasion	20	0.77 ^c	0.11		
Grinding	20	0.61 ^b	0.13		

*Different superscript letters in the same column show significant differences ($P<.05$).

Tukey HSD test of roughness data. All surface-treated specimens were rougher than the control group, and prominent differences were observed between the air-abraded and ground groups ($P<.005$). Air-abraded group had the roughest surface.

Table 2 summarizes the results of the Kruskal-Wallis and Mann-Whitney U tests of monoclinic content (%) values. Air-abraded or ground specimens showed higher monoclinic contents than control specimens ($P<.005$). However, in each surface treatment group, repeated firings did not affect the monoclinic content of the zirconia ($P>.05$).

According to strength analysis, two-way ANOVA test revealed that neither the surface treatments nor the firing affected the flexural strength of zirconia. On the other hand, Weibull analysis showed that in the air-abraded group only, repeated firings had a considerable influence on the characteristic strength. In this group, characteristic strength of the specimens was decreased significantly after 10 firing cycles as compared to that obtained after 2 firing cycles. In addition, all groups had a similar Weibull modulus, because confidence bounds for Weibull modulus overlapped ($P>.05$; Table 3).

A meaningful positive correlation between the rough-

ness and monoclinic content was observed ($P<.000$). However, no significant correlation was observed between the other pairs of values (either between the roughness and flexural strength or between the monoclinic content and flexural strength).

DISCUSSION

In clinical procedures, zirconia restorations are subjected to a series of steps including cutting, grinding, airborne-particle abrasion, firing of the veneering porcelain, and glazing. The surface treatments induce tetragonal-to-monoclinic phase transformation, whereas heat treatments induce reverse phase transformation (monoclinic to tetragonal). Hence, all of these procedures can cause stress concentration on the zirconia surface.¹⁶

Although the adaptation of the framework is performed in core trial, sometimes the inner surfaces of the restorations are adjusted minimally before glaze application,⁹ or surface treatments are applied before cementation to improve the bond between the resin cement and zirconia.²⁶ The surface treatments used in this study were chosen to mimic the preparation steps before cementation. As in pre-

Table 2. Results of the statistical analysis of monoclinic content values (%)

Surface treatment	Firings	N	Mean	Median	SD	Kruskal-Wallis test	
						Chi-square	P value
Control	2	10	1.91	2.03 ^a	0.20	41.132	.000
	10	10	2.08	2.03 ^a	0.22		
Air Abrasion	2	10	2.87	2.89 ^b	0.17		
	10	10	2.86	2.89 ^b	0.16		
Grinding	2	10	2.92	2.95 ^b	0.19		
	10	10	2.86	2.87 ^b	0.14		

*Different superscript letters in the same column show significant differences ($P<.05$).

Table 3. Mean, standard deviation, and Weibull statistics of flexural strength values (MPa)

Surface treatment	Firing cycles	Flexural strength				
		Mean (SD)	Shape (m)	95% CI	Scale (σ_0)	95% CI
Control	2	743.88 (231.81)	4.2	2.45-7.18	822.43 ^{ab}	704.94-959.49
	10	702.75 (236.80)	3.8	2.24-6.51	779.26 ^{ab}	658.24-922.52
Air Abrasion	2	756.93 (150.97)	6.1	3.71-9.90	816.43 ^b	732.83-909.56
	10	631.48 (71.97)	8.3	5.43-12.71	664.02 ^{ac}	613.24-719.0
Grinding	2	647.77(175.57)	4.2	2.65-6.81	712.27 ^{ab}	610.31-831.26
	10	700.38 (232.23)	3.6	2.21-5.93	778.34 ^{ab}	650.11-931.84
P value	>.05	.089		.024		

*Different superscript letters in the same column show significant differences ($P<.05$).

vious studies,^{17,27} layering with a veneering ceramic was omitted because the observed effects of the conditioning procedures on the framework material would be more complex if bilayer structures had been assessed. According to the statistical results on the monoclinic content data, the first hypothesis was partially rejected: only different surface treatments had an effect on the monoclinic phase content. After repeated firings, surface-treated specimens showed higher monoclinic contents than the control group ($P < .005$). Song *et al.*²⁷ reported that after heat treatment, all of the monoclinic content transformed to the tetragonal phase. On the contrary, Sato *et al.*¹⁶ showed that after firing between 800°C and 900°C, some monoclinic content remained on the surface. In this study, some negligible monoclinic content remained on the surface after the firing procedure, as was found by Sato *et al.*¹⁶ We think that whether monoclinic content remains depends on the material type. In addition, as in previous studies,²⁸⁻³⁰ the monoclinic phase content was significantly correlated with roughness values ($P < .000$).

Strength is an important parameter to determine the clinical success of the restoration.²⁵ It is affected by the sizes of flaws and defects on the material's surface, and it can be described as the maximum stress that is essential to cause fracture of a material.³¹ There are different testing methods available to assess the strength of ceramic materials: the three-point bending test,^{31,32,33} four-point bending test,²⁵ and biaxial flexural strength test.³⁴⁻³⁶ As in previous studies,^{3,11,32,33} the three-point bending test was used in this study, according to the ISO 6872:2008 standard.³⁷

Some authors^{11,16} reported that the increases in the strength of zirconia observed after surface treatments seemed to result mainly from the increase in monoclinic phase content. Other studies^{8,38} reported that subjecting a Y-TZP ceramic to various surface treatments resulted in distinct degrees of phase transformation (tetragonal to monoclinic), but statistically similar biaxial flexural strength values. On the other hand, Oh *et al.*³⁹ studied the effects of different firing techniques on the flexural strength of pressable all-ceramic materials and reported no noteworthy differences in the flexural strength values among the various groups. According to strength analysis, the second hypothesis was rejected that neither surface treatments nor repeated firings had an effect on flexural strength of zirconia ($P > .05$). However, according to Weibull analysis, the characteristic strength of zirconia was significantly decreased in the air-abrasion group after the 10th firing (Table 3). In general, the firing procedures did not have a negative effect on the strength of zirconia. However, we assume that in the air-abraded group, microcracks might be formed on the surface during surface treatment and after the 10th firing. This is because compared to the characteristic strength of the 2nd firing group, the characteristic strength of air-abraded zirconia fired 10 times was decreased significantly.

Based on these results, the clinician should be careful when working with zirconia, because surface treatment affects its monoclinic phase content, and depending on the

material type, a negligible amount of monoclinic content could be present on the surface after heat treatment. Under clinical conditions, multiple firings could be performed if necessary. However, it should not be forgotten that for rougher surfaces, the fracture probability could increase after multiple firings. The influence of loading under simulated oral conditions should be investigated in further studies to determine how the mechanical properties evolve under these conditions.

CONCLUSION

According to the findings of current study, after heat treatment, a negligible amount of monoclinic phase remained on zirconia surface. The monoclinic content of the zirconia was affected by surface treatments, and surface-treated ceramic specimens (air-abraded or ground) had higher monoclinic contents than the untreated (control) ones ($P < .005$). Surface treatments or multiple firings did not influence a decrease in flexural strength of zirconia, but for rougher surfaces, the failure probability could increase after multiple firings.

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