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Effect of sintering programs and surface treatments on monolithic zirconia

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PURPOSE. To investigate the effect of sintering programs and surface treatments on surface properties, phase transformation and flexural strength of monolithic zirconia. **MATERIALS AND METHODS.** Zirconia specimens were sintered using three distinct sintering programs [classic (C), speed (S), and superspeed (SS)] (n = 56, each). One sample from each group underwent scanning electron microscopy (SEM) and grain size analysis following sintering. Remaining samples were divided into five subgroups (n = 11) based on the surface treatments: control (CL), polish (P), glaze (G), grind + polish (GP), and grind + glaze (GG). One sample from each subgroup underwent SEM analysis. Remaining samples were thermally aged. Monoclinic phase volume, surface roughness, and three-point flexural strength were measured. Monoclinic phase volume and surface roughness were analyzed by Kruskal-Wallis and Dunn tests. Flexural strength was analyzed by two-way ANOVA and Weibull analysis. The relationships among the groups were analyzed using Spearman's correlation analysis. **RESULTS.** Sintering program, surface treatment, and sintering × surface treatment ($P \leq .010$) affected the monoclinic phase volume, whereas the type of surface treatment and sintering × surface treatment affected the surface roughness ($P < .001$). Type of sintering program or surface treatment did not affect the flexural strength. Weibull analysis revealed no significant differences between the m and σ_o values. Monoclinic phase volume was positively correlated with surface roughness in the SGG and SSP groups. **CONCLUSION.** After sintering monolithic zirconia in each of the three sintering programs, each of the surface treatments can be used. However, for surface quality and aging resistance, G or GG can be recommended as a surface finishing method. [J Adv Prosthodont 2024;16:25-37]

KEYWORDS
Flexural strength; Surface properties; Scanning electron microscopy; Aging; Zirconia

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INTRODUCTION

Yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics have been used to fabricate all-ceramic fixed prostheses, monolithic restorations, and implant abutments with the introduction of computer-aided design/computer-aided manufacturing (CAD-CAM) technology in dentistry. Monolithic Y-TZP restorations are characterized by their high biocompatibility, durability, esthetic properties, conservative tooth preparation, minimal wear on the opposing dentition, and low risk of porcelain chipping.[1] Despite their high compatibility, adjustments may be required before or after cementation.[2] The grinding process may result in roughening of the surface owing to the removal of the glaze or polishing layer. This leads to the formation of microcracks, a decrease in the resistance of the material, and wearing of the opposing tooth.[3] Therefore, the surface of the prostheses fabricated with zirconia must be smoothened before cementation of the restoration or after intraoral grinding processes. The use of several techniques, such as polishing or glazing, has been proposed after the adjustment of restorations.[4]

Depending on the temperature, zirconia exists in three different crystalline forms: monoclinic, tetragonal, and cubic. Monoclinic zirconia exists at temperatures lower than 1170°C. It transforms into tetragonal and cubic zirconia at 1170°C and 2370°C, respectively. However, the tetragonal phase is only partially stabilized.[5] Thus, factors such as grinding process,[6] heat generated during grinding, the mode and speed of grinding,[7] aging,[8-10] low temperatures or humidity,[11] temperature and pH changes, and cyclic loading during chewing[12] can affect the phase transformation.

Flexural strength measurements are frequently used to determine the strength of ceramic materials. Previous studies[13,14] have shown that the flexural strength of zirconia varies between 608 MPa - 1540 MPa, depending on the sintering parameters, surface treatments, and microstructure. Different surface treatments;[15-17] different surface treatments and low-temperature degradation;[18-20] sintering parameters;[21-26] sintering parameters and aging;[27-30] and sintering parameters, different surface treatments, and aging[31] have been reported to affect the microstructural properties, roughness, and mechanical strength of Y-TZP ceramics. Although different sintering programs, polishing, and glazing procedures are recommended by manufacturers as surface finishing methods for monolithic zirconia ceramics, the effects of different sintering programs and surface treatments on the surface topography, surface roughness, phase transformation, and flexural strength of monolithic zirconia remain unclear. Therefore, this *in vitro* study aimed to investigate the effect of sintering programs and surface treatments on surface properties, phase transformation and flexural strength of monolithic zirconia (3Y-TZP).

The research hypotheses were as follows: 1) The monoclinic phase volume (%) of monolithic 3Y-TZP ceramic would not be affected by the type of sintering program and surface treatment. 2) The surface roughness of monolithic 3Y-TZP ceramic would not be affected by the type of sintering program and surface treatment. 3) The flexural strength of monolithic 3Y-TZP ceramic would not be affected by the type of sintering program and surface treatment.

MATERIALS AND METHODS

Before designing this study, a power analysis was conducted to establish the sample size for each subgroup. The analysis, performed using G*power 3.1 software (Heinrich Heine University, Dusseldorf, Germany), revealed an effect size of 0.40 for numerical variables (monoclinic phase volume [%], surface roughness, and flexural strength data). With a type 1 error of 0.05 and a working power of 0.90, it was determined that a minimum of 9 samples was required in each subgroup. For the Weibull analysis, the minimum sample size was calculated to be 10, for a reliability of 0.80 and confidence level of 0.90. Based on these calculations, we decided to include 10 samples in each subgroup, resulting in a total of 150 samples, for statistical analyses.

A single type of colorless, partially sintered, monolithic, and translucent 3Y-TZP ceramic (inCoris TZI 55/19, Sirona, Bensheim, Germany) (55 × 19 × 15.5 mm) was used in this study. The 3Y-TZP ceramic was sectioned into bar shapes (25 × 5 × 2 mm) using a

low-speed sectioning machine (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) under water cooling (n = 168), considering a sintering shrinkage of 20%. All samples were cleaned ultrasonically (GB-928, Shantou Chuangxin Technology Co., Ltd., Shantou, China) for 5 min using distilled water. The samples were dried in an incubator (BINDER D-78532, BINDER GmbH, Tuttlingen, Germany) at 80°C for 30 min and stored in a zirconia ceramic-specific coloring liquid (A2) (inCoris TZI Coloring Liquid, Sirona, Bensheim, Germany) for 5 min. Before sintering, the colored samples were again dried in an etuv at 80°C for 30 min. The samples were then divided into three groups (n = 56) according to the sintering programs: classic (C), speed (S), and superspeed (SS). Sintering was performed in a zirconia sintering oven (inFire HTC speed, Sirona, Bensheim, Germany) according to the manufacturer's recommendations (Table 1).[32] The final dimensions of the samples were 20 × 4 × 1.6 mm. One sample from each group underwent scanning electron microscope (SEM) analysis (Nova NanoSEM 650, FEI Company, Hillsboro, SA, USA) (×50000) after sintering to evaluate the effect of the sintering program type on the surface topography and grain size of the zirconia. For grain size calculation, measurements were obtained from the widest regions of 30 randomly selected grains on the same SEM image, and the average grain size of each sample was calculated in nanometers (nm) and converted to micrometers (μm).

The remaining samples in each sintering group were divided into five subgroups according to the type of surface treatment (n = 11). No surface treatment was performed in the control (CL) subgroup. Disc-shaped green and orange rubbers (EVE DIACERA Ceramics Kit, EVE Ernst Vetter GmbH, Keltern, Germany) specific to this material were used in the polish (P) subgroup. One surface of the samples was polished using a handpiece at 7000 rpm for 30 s without water cooling with movements parallel and horizontal to the sample surface. Glaze material (Celtra Universal Overglaze, DeguDent GmbH, Hanau-Wolfgang, Germany) was applied to one surface of the samples in the glaze (G) subgroup, and glaze firing was performed in a porcelain oven (Programat P310, Ivoclar-Vivadent, Schaan, Liechtestein) in accordance with the manufacturer's recommendations. The glaze firing parameters were as follows: initial temperature (°C), 500; preheating time (min), 3.30; heating rate (°C/min), 60; final temperature (°C), 820; holding time (min), 1; and long-term cooling (°C), 750. In the grind + polish (GP) subgroup, first, one surface of the sample was ground at 8000 rpm using a dentin bur (EVE DIACERA Ceramics Kit, EVE Ernst Vetter GmbH, Keltern, Germany) for 30 s in one direction without water cooling, and subsequently the same procedure was repeated in the P subgroup. In the grind + glaze (GG) subgroup, first, one surface of the sample was ground at 8000 rpm using a dentin bur for 30 s in one direction without water cooling, and subsequently the same procedure was repeated in the G subgroup.

One sample from each of the sintering-surface treatment groups, yielding a total of 15 samples, was used for SEM analysis (×500) to observe the changes caused by the different surface treatments on the surface.

Table 1. Sintering parameters for each sintering program

Sintering Program Type	Sintering Parameters		
	Heating rate (°C/min)	Holding temperature (°C)	Holding time (min)
C	25	800	0
	15	1510	120
	30	200	0
S	99	750	0
	99	1100	0
	50	1510	30
	99	800	5
SS		1580	10

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To simulate one year of clinical use,[33] all surface-treated specimens (n = 150) were thermally aged (5°C - 55°C; 10000 cycles; duration, 30 s; and transfer time, 10 s) in a thermal cycling device (SD Mechatronik Thermocycler, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany). To determine the phase transformation of the zirconia ceramics, 150 samples were analyzed using an X-ray diffraction (XRD) device (PANalytical EMPYREAN, Malvern Panalytical, Almelo, The Netherlands). Using Cu as the radiation source, the treated surfaces of the samples were scanned with a 0.02 step interval at K-Alpha1 and K-Alpha2 wavelengths in the angle range of 2Θ 10° - 80°. The monoclinic phase volume (%) of zirconia was calculated for each sample using the following formulae:[34]

$$X_m = [I_m(-111) + I_m(111)] / [I_m(-111) + I_m(111) + I_t(101)];$$
$$V_m = 1.311 \times X_m / 1 + (0.311 \times X_m),$$

where X_m is the monoclinic peak intensity ratio; I_m (-111), I_m (111), and I_t (101) represent the intensity of the diffracted peaks in the monoclinic planes (-111) and (111) and the tetragonal plane (101); and V_m is the monoclinic phase volume content (%).

XRD analysis revealed that the tetragonal $_t$(101) peaks occurred around 30.14° - 30.41° 2Θ, whereas the monoclinic peaks of $_m$(-111) and $_m$(111) occurred around 28.12° - 31.46° and 31.09° - 31.96° 2Θ, respectively.

The surface roughness (Ra in μm) of all specimens was measured using a profilometer (TR200, TIME Group Inc, Beijing, China). Three measurements from different regions were obtained for each sample, and the average of these measurements was calculated. Subsequently, the three-point flexural strength test was performed at a loading rate of 1 mm/min using a universal testing machine (MOD DENTAL, Esetron, Ankara, Turkey) in accordance with ISO 6872:2008.[35] The load was applied to the center of the treated surfaces of each sample. The values at the time of fracture were recorded in "N" and the flexural strength values were calculated as "MPa" using the following formula:[36]

$$\text{Stress (MPa)} = 3Fd / 2wh^2,$$

where F is the fracture strength (N), d is the distance between the centers of the supports (mm), w is the width of the specimen (mm), and h is the height of the specimen (mm).

The results of the ''One-Sample Kolmogorov-Smirnov Test'' indicated that monoclinic phase volume (%) (P < .001) and surface roughness data (P < .001) did not demonstrate a normal distribution. However, the flexural strength data exhibited a normal distribution (P = .345).

The monoclinic phase volume (%) and surface roughness were analyzed using the Kruskal-Wallis and Dunn tests. The three-point flexural strength was analyzed using two-way analysis of variance (ANOVA) and Weibull analysis. Except Weibull analysis (Minitap 17 Program, State College, PA, USA), all other statistical analyses were performed using SPSS (version 23, IBM Corp., New York, USA). The Weibull distribution is defined using the following formula:[37]

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

where P is the fracture probability, σ is the flexural strength, σ_o is the characteristic strength value, and m is the Weibull modulus.

The relationship between the monoclinic phase volume (%) and surface roughness, monoclinic phase volume (%) and flexural strength, and surface roughness and flexural strength were analyzed using the Spearman correlation analysis (α = .05).

RESULTS

The SEM image (×50000) (Fig. 1) revealed smaller and irregular grain sizes, intergranular pores, and an increase in the number of small grains in the S and SS sintering groups. However, the grain boundaries could not be visualized clearly as the grains were stacked on top of each other. The average grain sizes of zirconia in the C, S, and SS groups were 0.36, 0.28, and 0.25 μm, respectively. The Figs. 2 - 4 present the SEM images (×500) of the sintered and surface-treated 3Y-TZP samples. Surface indentations were observed in the CL, P, and GP subgroups, whereas the surface was smooth in the G and GG subgroups. The surface structure became smoother from the C sintering to SS sintering groups in the P and GP groups.

The Kruskal-Wallis test revealed that the type of sintering program (P = .010), surface treatment type (P < .001), and sintering × surface treatment interac-

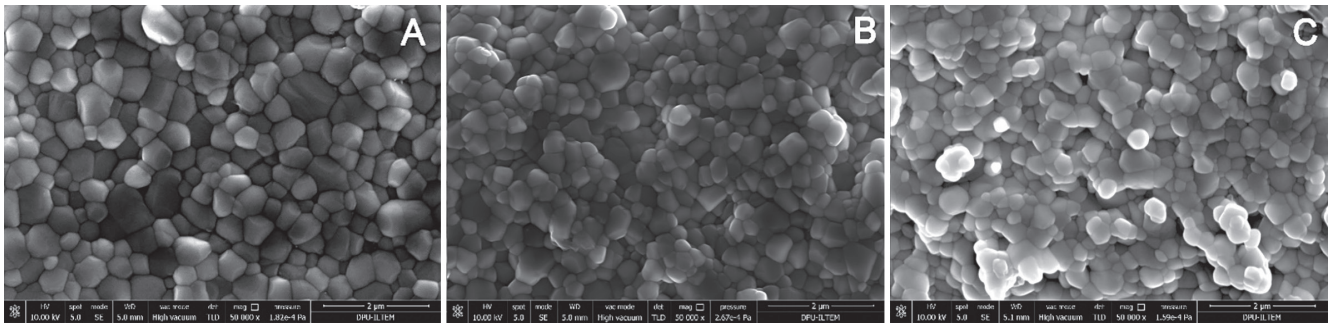
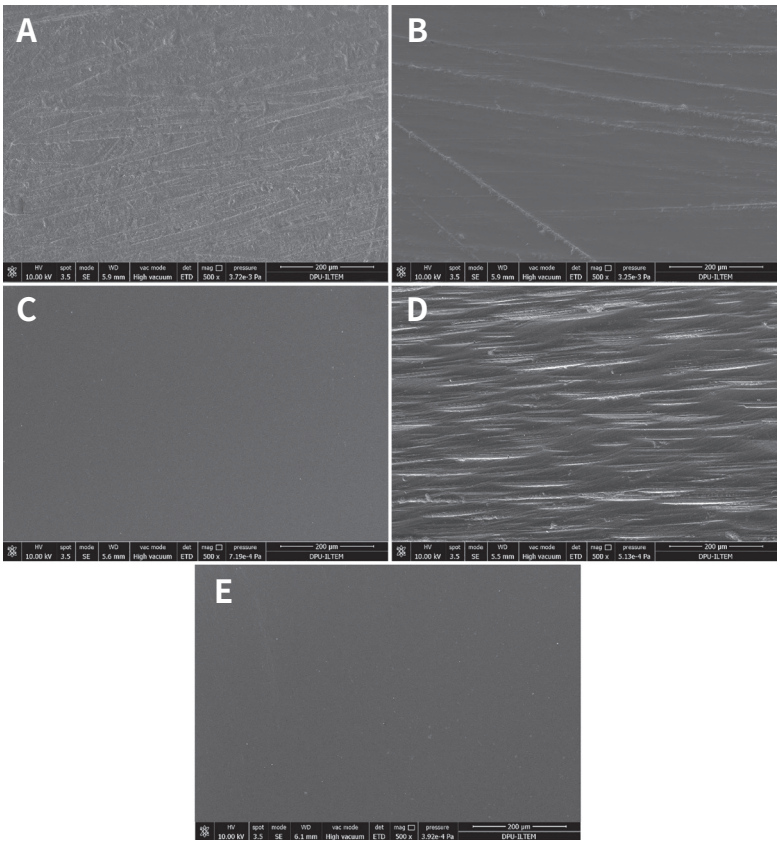


Fig. 1. SEM images (×50000) of zirconia sintered using different sintering programs. (A) Classic sintering; (B) Speed sintering; (C) Superspeed sintering.

Fig. 2. SEM images (×500) of the classic-sintered and surface-treated zirconia samples. (A) Control; (B) Polish; (C) Glaze; (D) Grind + Polish; (E) Grind + Glaze.



tion (P < .001) had an effect on the monoclinic phase volume (%) of zirconia. A significant difference was observed between the monoclinic phase volume (%) values of the C and SS groups (P = .007). Similarly, a significant difference was observed between the monoclinic phase volume (%) of the GP subgroup and those of the CL, P, G, and GG subgroups ($P \leq .002$). Comparison of all sintering × surface treatment groups revealed a significant difference between the CG and CGP groups, SCL and SGP groups, as well as SG and SGP groups ($P \leq .046$) (Table 2).

The Kruskal-Wallis test revealed that the surface treatment type (P < .001) and sintering × surface treatment interaction (P < .001) had an effect on the surface roughness. Significant differences were observed between the CL and G groups, CL and GP groups, CL and GG groups, P and G groups, P and GG groups, G and GP groups, as well as GP and GG

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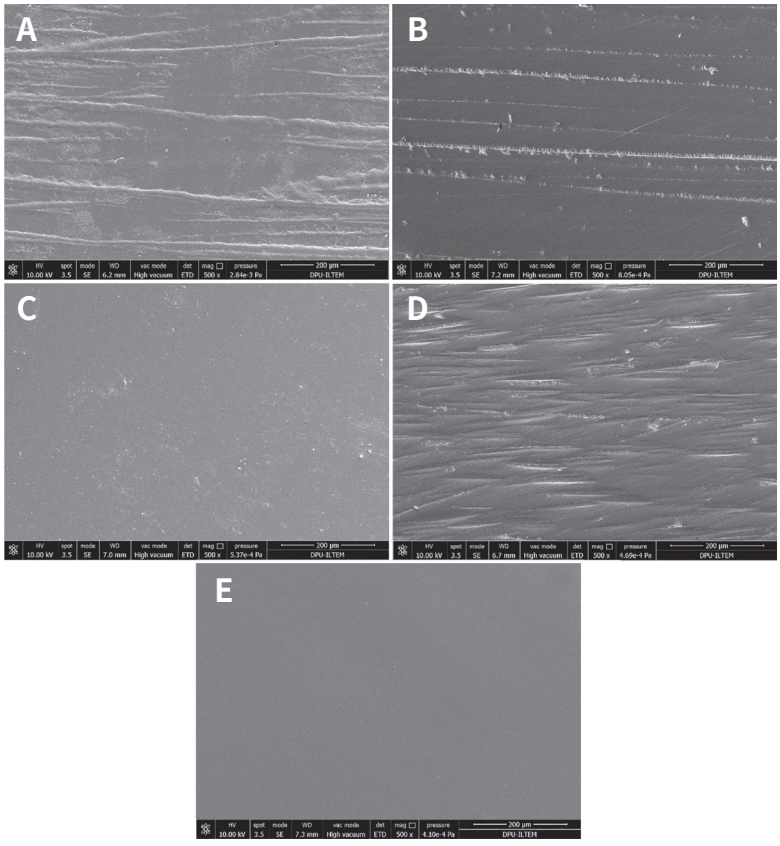

Fig. 3. SEM images (×500) of the speed-sintered and surface-treated zirconia samples. (A) Control; (B) Polish; (C) Glaze; (D) Grind + Polish; (E) Grind + Glaze.

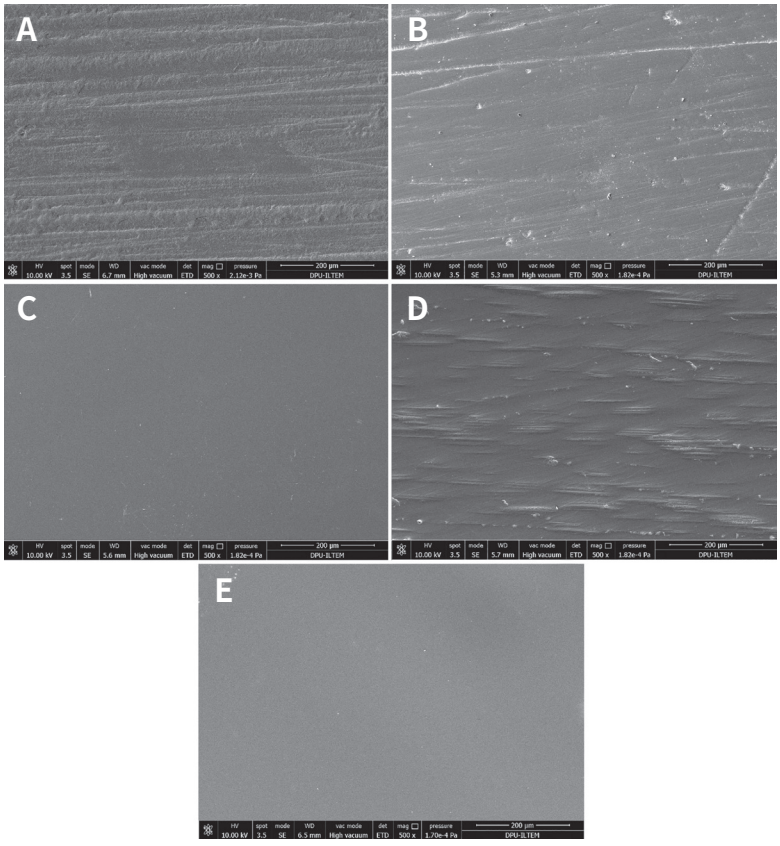

Fig. 4. SEM images (×500) of the superspeed-sintered and surface-treated zirconia samples. (A) Control; (B) Polish; (C) Glaze; (D) Grind + Polish; (E) Grind + Glaze.

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

Surface Treatment		Sintering Type C	Sintering Type S	Sintering Type SS	Total
CL	MN ± SD	0.90 ± 0.64	0.18 ± 0.38	0.20 ± 0.43	0.43 ± 0.59
	MD (Min-Max)	1.14^{ABC} (0-1.68)	0^{A} (0-0.92)	0^{A} (0-1.02)	0^{a} (0-1.68)
P	MN ± SD	2.01 ± 0.77	0.69 ± 0.90	0.65 ± 0.85	1.12 ± 1.03
	MD (Min-Max)	2.19^{ABC} (0-2.67)	0^{AB} (0-1.89)	0^{AB} (0-1.96)	1.52^{a} (0-2.67)
G	MN ± SD	0 ± 0	6.07 ± 13.02	0 ± 0	2.02 ± 7.81
	MD (Min-Max)	0^{A} (0-0)	0^{A} (0-35.51)	0^{A} (0-0)	0^{a} (0-35.51)
GP	MN ± SD	8.63 ± 1.07	6.56 ± 0.86	3.16 ± 2.76	6.12 ± 2.86
	MD (Min-Max)	8.81^{C} (6.56-9.77)	6.32^{BC} (5.75-8.13)	4.72^{ABC} (0-6.27)	6.37^{b} (0-9.77)
GG	MN ± SD	7.07 ± 11.55	6.17 ± 10.30	11.46 ± 24.17	8.23 ± 16.16
	MD (Min-Max)	0^{ABC} (0-28.21)	0^{AB} (0-26.86)	0^{AB} (0-58.25)	0^{a} (0-58.25)
Total	MN ± SD	3.72 ± 6.10	3.93 ± 7.70	3.10 ± 11.31	
	MD (Min-Max)	1.29^{1} (0-28.21)	0^{12} (0-35.51)	0^{2} (0-58.25)	

* MN ± SD: Mean ± Standard Deviation, MD (Min-Max): Median (Minimum-Maximum).
** The same capital letters indicate no difference between sintering × surface treatment type interaction, whereas the same small letters in the same column indicate no difference between the surface treatment groups. The same numbers in the same row indicate no difference between the type of sintering program.

Table 3. Results of the statistical analysis of roughness values (μm)

Surface Treatment		Sintering Type C	Sintering Type S	Sintering Type SS	Total
CL	MN ± SD	1.56 ± 0.55	1.33 ± 0.29	1.30 ± 0.33	1.40 ± 0.41
	MD (Min-Max)	1.53^{A} (0.63-2.28)	1.29^{A} (0.87-1.72)	1.26^{A} (0.77-1.86)	1.35^{a} (0.63-2.28)
P	MN ± SD	1.24 ± 0.41	0.73 ± 0.27	0.95 ± 0.35	0.97 ± 0.40
	MD (Min-Max)	1.25^{A} (0.68-1.88)	0.75^{ABC} (0.38-1.26)	0.84^{AC} (0.63-1.52)	0.83^{ac} (0.38-1.88)
G	MN ± SD	0.47 ± 0.15	0.52 ± 0.17	0.45 ± 0.05	0.48 ± 0.13
	MD (Min-Max)	0.52^{BC} (0.23-0.64)	0.51^{BC} (0.30-0.74)	0.45^{BD} (0.34-0.52)	0.49^{b} (0.23-0.74)
GP	MN ± SD	0.77 ± 0.12	0.64 ± 0.14	0.77 ± 0.09	0.72 ± 0.13
	MD (Min-Max)	0.76^{ACD} (0.59-1.00)	0.59^{AB} (0.48-0.86)	0.79^{AB} (0.62-0.87)	0.76^{c} (0.48-1.00)
GG	MN ± SD	0.44 ± 0.10	0.47 ± 0.14	0.44 ± 0.17	0.45 ± 0.13
	MD (Min-Max)	0.41^{BC} (0.32-0.58)	0.50^{BC} (0.29-0.71)	0.42^{BD} (0.19-0.80)	0.44^{b} (0.19-0.80)
Total	MN ± SD	0.89 ± 0.54	0.74 ± 0.37	0.78 ± 0.40	
	MD (Min-Max)	0.71^{1} (0.23-2.28)	0.63^{1} (0.29-1.72)	0.68^{1} (0.19-1.86)	

* MN ± SD: Mean ± Standard Deviation, MD (Min-Max): Median (Minimum-Maximum).
** The same capital letters indicate no difference between sintering × surface treatment type interaction, whereas the same small letters in the same column indicate no difference between the surface treatment groups. The same numbers in the same row indicate no difference between the type of sintering program.

groups in terms of surface treatment type ($P \leq .001$). Comparison of the surface roughness for each type of sintering program revealed significant differences between the CCL and CG groups, CCL and CGG groups, CP and CG groups, CP and CGG groups, SCL and SG groups, SCL and SGG groups, SSCL and SSG groups, SSCL and SSGG groups, SSP and SSG groups, as well as SSP and SSGG groups in terms of the surface treatments ($P \leq .048$) (Table 3).

Two-way ANOVA revealed that the type of sintering program ($P = .125$), surface treatment type ($P = .135$), and sintering × surface treatment interactions

(*P* = .403) had no significant effect on the three-point flexural strength. Weibull analysis (Table 4) revealed no difference when the m and $\sigma_o$values of all groups were compared among themselves (*P* > .05).

A significant positive correlation was observed between the surface roughness and monoclinic phase volume (%) in the SGG (r = .674, *P* = .033) and SSP (r = .747, *P* = .013) groups. No significant correlation was observed between the surface roughness and flexural strength and the flexural strength and monoclinic phase volume (%) of all groups (Table 5).

DISCUSSION

The first and second hypotheses were rejected as the monoclinic phase volume (%) was affected by the type of sintering program (*P* = .010) and surface treatment (*P* < .001), whereas surface roughness was only affected by the surface treatment type (*P* < .001). The third hypothesis was accepted as three-point flexural strength was not affected by the types of sintering program and surface treatment.

The effects of both sintering temperature and time[29,38] and only sintering temperature[39,40] on the grain size of zirconia ceramics (3Y-TZP) were investigated. Researchers stated that larger grain sizes were observed in classic sintering compared to speed and/or superspeed sintering.[29,38] Superspeed sintered 5Y-TZP[29] or 3Y-TZP[38] reportedly exhibited smaller average grain sizes than classic- and speed-sintered samples. Researchers who only examined the effect of different sintering temperatures observed that the grain size of zirconia (3Y-TZP, 4Y-TZP, and 5Y-TZP) increased as the sintering temperature increased.[39,40] They noted that temperatures above 1550°C led to the formation of holes in the microstructure of sintered zirconia, grain ruptures, and accumulation at grain boundaries.[39,40] In this study, SEM analysis was performed to determine the effect of the type of sintering program on the grain size of zirconia and interpret the effect of different surface treatments on the surface topography of sintered zirconia. Similar to previous studies, the zirconia grain size in this study increased with decreasing sintering temperature or increasing holding time at the same sintering temperature.[29,38] Additionally, as observed by previous studies, SS sin-

Table 4. Mean, standard deviation, and results of the Weibull analysis of flexural strength (MPa)

Group (Sintering-Surface Treatment)	Mean (SD)	m (SE)	m %95 CI	σ_o (SE)	σ_o%95 CI
CCL	652.61 (148.61)	5.16 (1.26)	3.20-8.31	709.30 (45.94)	624.74-805.30
CP	659.62 (111.81)	7.22 (1.86)	4.35-11.97	705.52 (32.65)	644.34-772.50
CG	717.01 (140.80)	6.79 (1.80)	4.03-11.40	770.94 (37.67)	700.54-848.41
CGP	712.45 (87.69)	7.82 (1.71)	5.08-12.00	751.65 (32.41)	690.74-817.92
CGG	613.59 (153.71)	5.95 (1.65)	3.45-10.25	663.01 (36.28)	595.58-738.06
SCL	668.15 (167.75)	5.90 (1.68)	3.37-10.30	725.55 (40.22)	650.84-808.82
SP	595.25 (137.62)	5.50 (1.40)	3.33-9.07	646.18 (38.97)	574.13-727.25
SG	534.33 (209.47)	2.98 (0.82)	1.73-5.12	592.99 (64.96)	478.40-735.02
SGP	691.58 (79.36)	9.89 (2.37)	6.7-15.82	725.93 (24.61)	679.25-775.79
SGG	618.19 (127.21)	6.28 (1.66)	3.73-10.55	667.47 (35.35)	601.66-740.47
SSCL	608.43 (117.72)	5.31 (1.19)	3.42-8.23	656.39 (41.59)	579.73-743.18
SSP	588.15 (149.53)	4.56 (1.12)	2.81-7.37	644.57 (47.34)	558.15-744.37
SSG	658.89 (140.78)	6.93 (1.91)	4.04-11.88	708.18 (33.49)	645.48-776.96
SSGP	671.63 (132.89)	6.41 (1.61)	3.91-10.47	722.17 (37.45)	652.36-799.44
SSGG	586.86 (103.72)	6.37 (1.50)	4.01-10.11	628.91 (33.08)	567.31-697.20
P value		.352		.102	

SD: Standard Deviation, m: Weibull Modulus, SE: Standard Error, σ_o: Characteristic Strength Value, CI: Confidence Interval.

Table 5. Results of the correlation analysis

Group (Sintering-Surface Treatment)		Roughness-Monoclinic Phase Volume (%)	Roughness-Flexural Strength	Flexural Strength-Monoclinic Phase Volume (%)
CCL	r	-.080	-.442	-.448
	P	.827	.200	.194
CP	r	.127	.382	.491
	P	.726	.276	.150
CG	r	–	-.523	–
	P	–	.121	–
CGP	r	-.146	-.220	.018
	P	.687	.542	.960
CGG	r	.007	-.212	.231
	P	.984	.556	.521
SCL	r	-.447	-.292	-.043
	P	.195	.413	.906
SP	r	.102	-.527	-.055
	P	.778	.117	.881
SG	r	-.030	.250	.588
	P	.933	.486	.074
SGP	r	.358	-.273	.176
	P	.310	.446	.627
SGG	r	.674	.209	.425
	P	.033*	.562	.221
SSCL	r	.161	-.043	.588
	P	.658	.907	.074
SSP	r	.747	-.103	-.068
	P	.013*	.776	.851
SSG	r	–	-.624	–
	P	–	.054	–
SSGP	r	.386	.055	.319
	P	.271	.881	.369
SSGG	r	-.356	.304	.588
	P	.313	.393	.074

* $P < .05$, r: Correlation Coefficient, *P*: *P* value.

tered (1580°C) zirconia showed an increase in the number of holes and substantial grain agglomeration (Fig. 1).[39,40] The surface became smoother from C sintering to SS sintering, particularly in the P and GP subgroups (Fig. 2, Fig. 3, and Fig. 4). The higher sintering temperature (1580°C) of the SS sintering program, compared with that of the sintering temperature (1510°C) of the C and S sintering programs, may have had an effect on the surface.

The effects of the surface treatments and aging[18-20] or only aging[28,29,41-44] on the phase transformation of zirconia were investigated. Although the content of monoclinic zirconia varied depending on the type of surface treatment,[18,20] sintering protocol, yttria content, material type, and aging,[19,28,29,41-44] in this study the monoclinic phase volume (%) after aging varied according to the type of sintering program and surface treatment. Although the monoclinic phase was not observed in the CG and SSG groups, it was observed in the SG group. For the SG group, the short holding

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time (30 min) and high cooling rate (99°C/min) during speed-sintering may have caused the aging resistance of the material to be low. Additionally, the absence of the monoclinic phase in the CG and SSG groups may be attributable to the longer sintering time used in C sintering and the high sintering temperature (1580°C) used in SS sintering.

The effects of the sintering parameters[31,42,45,46] and surface treatments[31] on the surface roughness of zirconia (3Y-TZP) were also investigated. While it was stated that sintering temperature[42,45,46] and/or time[42,45] did not significantly affect the surface roughness values, Hafezeqoran *et al*.[31] reported that surface roughness values were influenced by the sintering factor, but not by the surface treatment factor. Furthermore, they reported that speed sintering provided significantly smoother surface structure than classic sintering.[31] Similar to the findings of previous studies,[42,45,46] the surface roughness was not affected by the sintering temperature and duration in this study; however, the findings of this study varied from those of the study by Hafezeqoran *et al*.,[31] in which the surface roughness was affected by the surface treatment type ($P < .001$).

Material durability plays an important role in the evaluation of the long-term clinical success of dental ceramics. Several flexural strength tests (three-point, four-point, and biaxial) have been proposed by ISO 6872:2008 to evaluate the strength of ceramic materials.[35] The flexural strength of dental ceramics should be more than 300 MPa for single-unit restorations and more than 500 MPa for 4-unit prostheses.[35] The flexural strength values (MPa) of all groups met these specifications in this study.

The effects of the sintering parameters[27-30,41,42] and different hydrothermal degradation protocols[28,41,44,47-50] on the strength of zirconia were investigated. Previous studies[27,30] have demonstrated a decrease in the flexural strength. However, Kong and Park[28] reported that the flexural strength increased after 12 h of sintering. Other studies[41,42] have reported that the sintering temperature and duration did not affect the flexural strength. Liu *et al*.[29] reported that the biaxial flexural strength of classic- and speed-sintered zirconia (4Y-TZP, 5Y-TZP, and 6Y-TZP) were similar, whereas superspeed-sintered 5Y-TZP had higher flexural strength than those of the classic- and speed-sintered samples. Similar to the findings of previous studies,[41,42] the flexural strength values were not affected by the sintering temperature or duration in this study. The difference between the flexural strength observed in this study and those observed in other studies[27-30] may be due to the differences in the formulation of the ceramic materials, sintering parameters, and flexural strength test type. In this study, since the sintering programs used were those recommended by the manufacturer[32] and no significant correlation was observed between flexural strength and monoclinic phase volume data, the flexural strength values of the zirconia remained unaffected by the sintering temperature and time.

This study had certain limitations. A single type of partially sintered monolithic 3Y-TZP ceramic was used in the *in vitro* analyses, and only thermal aging was performed. The phase transformation, roughness, and three-point flexural strength were assessed only after aging following the surface treatment. In future studies, the effects of different sintering parameters and surface treatments on the color, microstructure, physical, and mechanical properties of different types of monolithic zirconia ceramics should be investigated both before and after aging. Further long-term clinical studies are essential to obtain reliable results.

CONCLUSION

Bearing in mind the limitations of this *in vitro* study, the following conclusions can be drawn:

The grain sizes of monolithic zirconia sintered using different programs were affected by the sintering temperature and holding time. The grain sizes of the zirconia decreased as the sintering temperature increased or as the holding time decreased at the same sintering temperature.

According to the correlation analysis results in the SGG and SSP groups, significant positive correlations were found between roughness-monoclinic phase volume (%) data. In clinical practice, surface treatments on monolithic zirconia ceramics should be performed carefully because surface treatments can cause phase transformations.

Based on the results of SEM, XRD, surface rough-

ness, and flexural strength analyses, after sintering monolithic zirconia in each of three sintering programs, each of surface treatments can be used. However, for surface quality and aging resistance, G or GG can be recommended as a surface finishing method.

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