Effect of different grinding burs on the physical properties of zirconia

Kyung-Rok Lee[1], Han-Cheol Choe[2], Yu-Ri Heo[1], Jang-Jae Lee[3], Mee-Kyoung Son[1]*

[1]Department of Prosthodontics, School of Dentistry, Chosun University, Gwangju, Republic of Korea
[2]Department of Dental Materials, School of Dentistry, Chosun University, Gwangju, Republic of Korea
[3]Department of Computer Science and Statistics, College of Natural Sciences, Chosun University, Gwangju, Republic of Korea

PURPOSE. Grinding with less stress on 3Y-TZP through proper selection of methods and instruments can lead to a long-term success of prosthesis. The purpose of this study was to compare the phase transformation and physical properties after zirconia surface grinding with 3 different grinding burs. **MATERIALS AND METHODS.** Forty disc-shaped zirconia specimens were fabricated. Each Ten specimens were ground with AllCeramic SuperMax (NTI, Kahla, Germany), Dura-Green DIA (Shofu Inc., Kyoto, Japan), and Dura-Green (Shofu Inc., Kyoto, Japan). Ten specimens were not ground and used as a control group. After the specimen grinding, XRD analysis, surface roughness test, FE-SEM imaging, and biaxial flexural strength test were performed. **RESULTS.** After surface grinding, small amount of monoclinic phase in all experimental groups was observed. The phase change was higher in specimens, which were ground with Dura-Green DIA and AllCeramic SuperMax burs. The roughness of surfaces increased in specimens, which were ground with Dura-Green DIA and AllCeramic SuperMax burs than control groups and ground with Dura-Green. All experimental groups showed lower flexural strength than control group, but there was no statistically significant difference between control group and ground with Dura-Green DIA and AllCeramic SuperMax burs. The specimens, which were ground with Dura-Green showed the lowest strength. **CONCLUSION.** The use of dedicated zirconia-specific grinding burs such as Dura-Green DIA and AllCeramic SuperMax burs decreases the grinding time and did not significantly affect the flexural strength of zirconia, and therefore, they may be recommended. However, a fine polishing process should be accompanied to reduce the surface roughness after grinding. *[J Adv Prosthodont 2016;8:137-43]*

KEY WORDS: 3Y-TZP; Phase transformation; XRD; Roughness; Biaxial flexural strength

INTRODUCTION

Zirconia exhibits superior mechanical properties and biocompatibility, and has been widely used in dentistry for implant frameworks, crowns, bridges, orthodontic material.[1,2] Ever since the publication of an article in Nature entitled "Ceramic steel?",[3] the research activities on Zirconia have grown steadily.

Zirconia typically exhibits three polymorphisms: monoclinic, tetragonal, and cubic phases. In addition, the orthorhombic form also existed at high pressure, and the rhombohedral phase produced in abraded surfaces of zirconia after grinding.[3,4] At room temperature, pure zirconia exists in the monoclinic phase; however, as the temperature increases, the monoclinic phase transforms to the tetragonal phase. Further, at a temperature > 1170°C, the tetragonal phase becomes stable.[5] After high-temperature sintering, while cooling the zirconia to the room temperature, the tetragonal phase transforms back to the monoclinic phase, and its volume increases by 3 - 5%. The resulting strain exceeds the elasticity and fracture strength of zirconia, forming cracks in the zirconia.[1] The addition of metal oxides such as MgO, CaO, or T_2O_3 to pure zirconia lowers the phase-transition (tetragonal to monoclinic) temperature to below

Corresponding author:
Mee-Kyoung Son
Department of Prosthodontics, School of Dentistry, Chosun University, 309 Pilmun-daero, Dong-gu, Gwangju 61452, Republic of Korea
Tel. 82 622203825: e-mail, son0513@chosun.ac.kr

ambient temperature, thereby allowing the zirconia to exist in the tetragonal phase at ambient temperature. This process is known as metastabilization.[1] The metastable zirconia is also known as tetragonal zirconia polycrystal (TZP), and 3Y-TZP, i.e., TZP containing 3% Y_2O_3, is commonly used currently.

The high toughness and strength of TZP arises from the tetragonal-to-monoclinic phase-transformation toughening of the stress region under the load. The phase change absorbs the fracture energy of the crack line, thus preventing the progression of the crack. Furthermore, the volume increase due to the phase change is suppressed by the tetragonal phase, which results in compression stress on the crack line, thereby leading to an increase in tension.[1,5,6] However, excessive phase changes and corresponding volume increase may cause cracks and a decrease in flexural strength. Several factors such as the metastability of the tetragonal phase, heat generated from grinding or polishing, and polishing strength influence the phase change in TZP, in a complex manner.[7-11] It has been reported that flaws or stress layers may be formed when grinding or polishing is conducted on a zirconia surface, and in severe cases, an increase in the size of the flaw or a profound penetration of stress leads to a decrease in flexural strength.[2,5]

Zirconia prostheses require a grinding process in order to increase its adaptability or to adjust the occlusion during the fabrication or the clinical application stage.[11-13] Silicone green stone has been frequently used for grinding and polishing of conventional ceramic or composite material; however, for grinding zirconia, it is recommended that dedicated zirconia-specific grinding apparatuses and materials be used. These include a conventional silicone stone with a diamond grit implanted on it or apparatuses made of a ceramic material, leading to higher grinding efficiency and less heat generation. Although many dedicated zirconia-specific polishing apparatuses to increase the grinding efficiency of zirconia and reduce strain have been developed and recommended, studies on the changes in the properties of zirconia using these apparatuses have not been conducted.

Therefore, the goal of this study was to compare and assess the phase changes, changes in surface roughness, and changes in flexural strength of zirconia using three different grinding burs.

MATERIALS AND METHODS

Thirty disc-shaped specimens (14 mm diameter, 1.4 mm thickness) were prepared using a 3Y-TZP zirconia block (Prettau, Zirkonzahn, Italy). Ten specimens were randomly grouped into each of the three groups of different grinding burs. Groups A, B, and C were ground using AllCeramic SuperMax (G8002, NTI, Kahla, Germany), Dura-Green DIA (PN0155, Shofu Inc., Kyoto, Japan), and Dura-Green (PN0042, Shofu Inc., Kyoto, Japan) (Fig. 1). AllCermic SuperMax and Dura-Green DIA are commonly used as dedicated zirconia-specific grinding burs, however Dura-Green is used for grinding of conventional ceramic restoration.
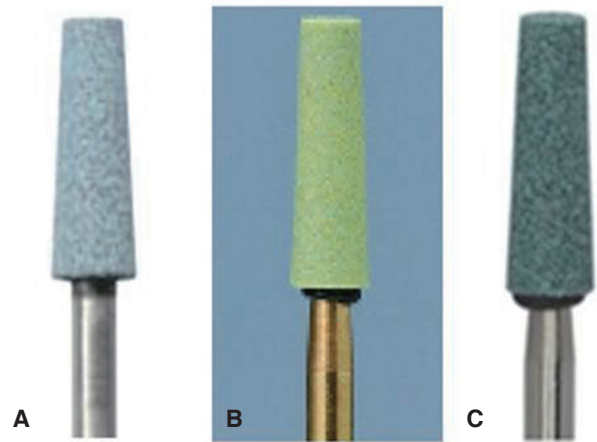

Fig. 1. Grinding burs used in this study. (A) NTI® AllCeramic SuperMax, (B) Shofu Dura-Green DIA, (C) Shofu Dura-Green.

The specimens in each experimental group were ground using a Micromotor (NSK ultimate 500, Japan) at 20,000 rpm. A cylindrical shape grinding burs were used, and each specimens were uniformly ground around 0.15 ± 0.03 mm. The weight of specimens before grinding was 1.30 ± 0.02 g, and 0.18 ± 0.01 g was uniformly ground by grinding. Additional ten disk-shaped specimens without grinding (14 mm diameter, 1.25 mm thickness) were prepared and set as a control group.

The crystallographic phase changes in the three experimental and control groups were observed using an X-ray diffractometer (X'pert powder, PANalytical, Almelo, Netherlands). Radiation condition was as follows: Cu Kα radiation, 40 kV, 30 mA, 0.03°/step, 27 - 65 θ range. After grinding of the specimen surfaces, the changes in the relative monoclinic phase fraction (X_m) were calculated by using a method reported by Garvie and Nicholson.[14] This is the most commonly used method to determine the amount of phase change from the tetragonal to monoclinic phase, and the changes are calculated as follows:

$$X_m = (I_{-(111)m} + I_{(111)m}) / (I_{-(111)m} + I_{(111)m} + I_{(111)t})$$

(I: Integral intensity at 2θ, (111)t: Tetragonal peak, -(111)m & (111)m: main peak of Monoclinic)

The transformed zone depth (TZD), i.e., the depth of phase change from the surface, was calculated by using a method reported by Kosmac et al.[15]

TZD (μm) = sinθ/2μ ln(1/1-X_m), (θ = angle of reflection 15°, μ = absorption coefficient 0.0642)

As the rate of change from the tetragonal to monoclinic phase increases, the asymmetry and width of the (111)t

peak increases, and these parameters were quantified by calculating and assessing H (Δ).[6]

H (Δ) = Full peak width at half maximum

The surface roughness values were measured using a Surfcorder SE1700 rugosimeter (Kosaka Laboratory Ltd., Kosaka, Japan), and ten tests were performed for each group. The experimental conditions were 0.5 mm/s velocity and 4 mm distance, using a 5 µm diameter tip.

The surface microstructure was analyzed using a field emission scanning electron microscope (FE-SEM). After grinding the specimens, the surfaces of the grinding burs and the specimen surfaces were observed at 500× and 1000× magnifications.

The biaxial flexural strengths of the specimens were measured through a biaxial flexural strength experiment using a piston and three ball supports, according to the international standard ISO 6872 for dental ceramic materials.[16] Three balls with 3.4 mm diameter each were placed at a 120° angle in an equilateral triangle on a support circle of 10 mm diameter. Using a universal testing machine (DSC-500, Shimadzu Corp., Kyoto, Japan), a piston with a flat circular surface of 1.4 mm diameter was used to apply a load of 500 g. The crosshead speed was set at 0.5 mm/min (Fig. 2). To distribute load, a 0.05 mm plastic sheet was placed. After the maximum fracture strength (N) was recorded, the flexural strength (S) was calculated by using the Poisson's ratio (0.25) as follows.[16]

$$S = -0.2387P(X - Y) / d^2$$
$$X: (1 + \upsilon) \ln(r_2 / r_3)^2 + [(1 - \upsilon) / 2] (r_2 / r_3)$$
$$Y: (1 + \upsilon) [1 + \ln(r_1 / r_3)^2] + (1 - \upsilon) (r_1 / r_3)^2$$

(S: Maximum tensile stress in MPa, P: Total load causing fracture in N

υ: Poisson's ratio, r_1: the radius of the support circle (mm), r_2: the radius of the loaded area (mm), r_3: the radius of the specimen (mm), d: the specimen thickness at fracture origin (mm))

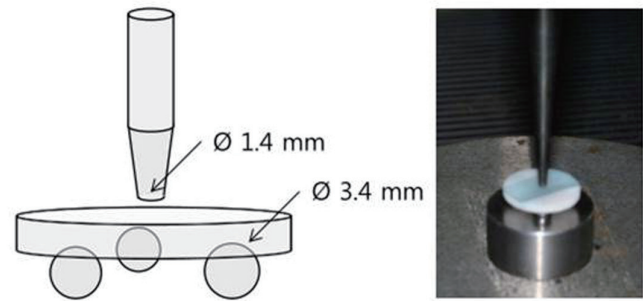The statistical analysis was performed to compare five different test values of four independent groups. First, the normality and homogeneity of variance of the four independent groups was tested. For five test results, Welch's ANOVA test was used because all test value of four independent groups satisfied the assumption of the normality, but violated the homogeneity of variance. As a post-hoc test, Games-Howell test was performed. Two tailed P value was calculated, and less than 0.05 was analyzed as statistically significant (P < .05).

RESULTS

X_m and TZD values showed statistically significant difference between four groups in order Groups B > A > C > Control (P < .001) (Table 1). H (Δ) was calculated and assessed based on the following property: the pure the crystallographic phase is, the narrower and higher the profile of the peak is, and as the phase changes, the profile widens and shows asymmetry. The H (Δ) values at the (111)t peak of the tetragonal phase showed statistically significant difference between four groups in order Groups B > A > C > Control (P < .001) (Table 1). The control group exhibited 100% tetragonal phase. After grinding the surface, an increase in the monoclinic phase in all the three experimental groups was observed. In particular, the phase change was higher in Groups A and B, where dedicated zirconia-specific grinding burs were used.

Fig. 2. Biaxial flexural strength testing apparatus: ISO Standard 6872 for dental ceramic.

Table 1. Result of XRD analysis. Relative amount of monoclinic zirconia (Xm: %), TZD (µm) and H (Δ) for groups

Groups	N	Xm (%)	TZD (µm)	H (Δ)
Control	10	0.00 ± 0.00*	0.00 ± 0.00†	0.00 ± 0.00‡
A	10	4.77 ± 0.37*	0.10 ± 0.01†	0.48 ± 0.04‡
B	10	5.99 ± 0.31*	0.12 ± 0.01†	0.55 ± 0.06‡
C	10	3.25 ± 0.58*	0.07 ± 0.01†	0.39 ± 0.06‡

Values are Mean ± Standard deviation.

*, †, ‡ indicate the significance of differences between the test groups (P < .001).

Table 2. The mean roughness values for groups (µm)

Groups	N	Mean ± SD	P value
Control	8	0.97 ± 0.24[a]	
A	8	1.87 ± 0.41[b]	
B	8	1.82 ± 0.18[b]	< .001
C	8	1.02 ± 0.07[a]	

Different superscripts indicate the significance of differences between the test groups.

In the analysis of surface roughness, the roughness of surfaces increased in Groups A and B, where dedicated zirconia-specific grinding burs were used than Control group and Group C (Table 2).

According to the manufacturer's introduction, AllCeramic SuperMax bur includes sintered diamond with ceramic bonding for cutting on ceramic, aluminous oxide and zirconium. To obtain the best cutting efficiency, this bur includes a mixture of diamonds. Dura-Green DIA bur includes densely packed diamonds in a special glass binder offering extended cutting ability for ceramic, zirconia and hard alloys. Dura-Green bur is impregnated with silicon carbide grains in a special glass binder for cutting on composite, ceramic and alloys. The microstructures of grinding burs were analyzed using FE-SEM. AllCeramic SuperMax bur showed a typical ceramic surface possibly made by sintering particles. Density of apparatus and presence of porosities affect the grinding efficiency and the surface roughness, and this bur had a high density and few porosities. Dura-Green DIA bur increased the grinding efficiency by mixing diamond particles. Dura-Green bur showed a typical ceramic surface, and did not contain diamond particles that could have increased the grinding efficiency, and this bur seems to exhibit the lowest grinding efficiency (Fig. 3).

The microstructures of the specimen surface after grinding were analyzed using FE-SEM. The surface of control group showed unadulterated sintered crystals. Group A showed a rough surface because of mechanical grinding. The irregular surface of the grinding bur made of sintered ceramic seems to have contributed to the rough surface of the specimen. In Group B, the diamond grit seems to have increased the grinding efficiency and roughness. Group C showed smaller scratch grooves and a smoother surface, compared to Groups A and B (Fig. 4).
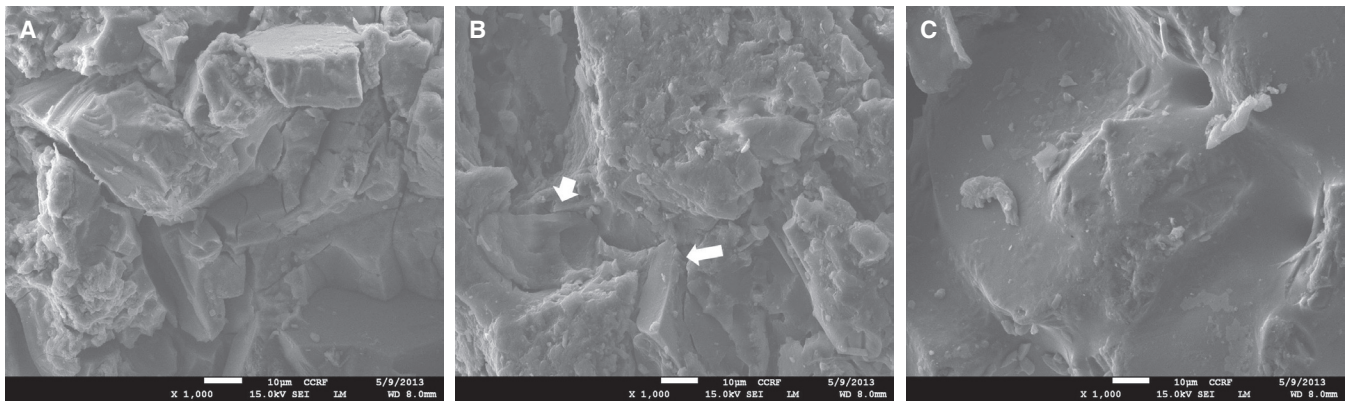

Fig. 3. FE-SEM images of bur surface (×1000) after specimen grinding. (A) NTI® AllCeramic SuperMax, (B) Shofu Dura-Green DIA, (C) Shofu Dura-Green. The arrow points indicate diamond grit.
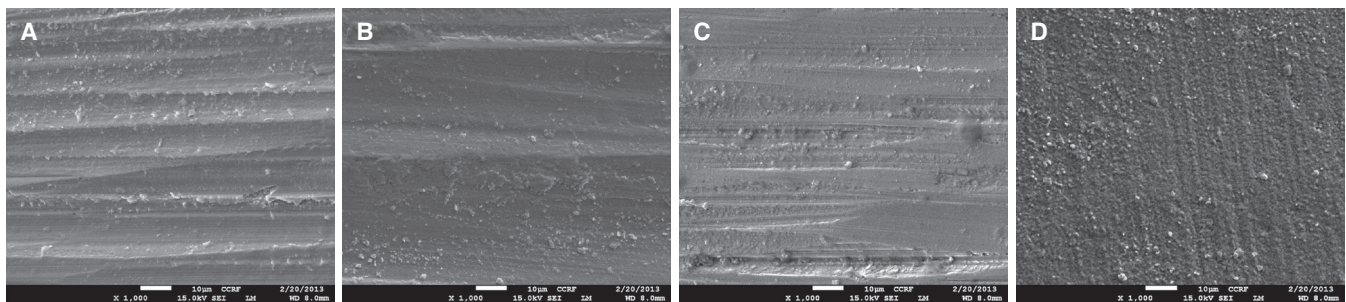

Fig. 4. FE-SEM images of the specimen surface (×1000). (A) Group A, (B) Group B, (C) Group C, (D) Control Group. Group C showed smaller scratch grooves and a smoother surface, compared to Groups A and B.

Table 3. The mean biaxial flexural strength values (MPa) for groups

Group	N	Mean ± SD	P value
Control	10	945.86 ± 49.05[a]	
A	10	870.50 ± 101.60[a]	
B	10	815.54 ± 149.84[ab]	< .001
C	10	670.16 ± 103.42[b]	

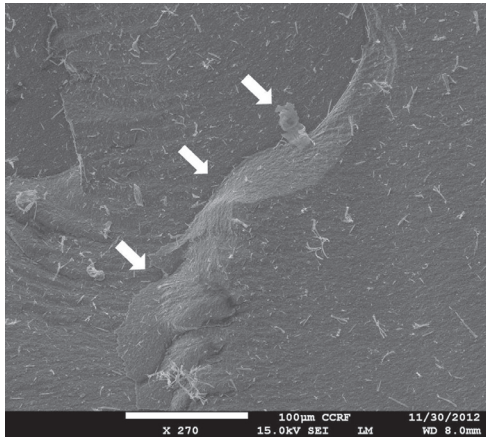Different superscripts indicate the significance of differences between the test groups.

Fig. 5. FE-SEM images of the fracture surface in experimental group specimen after flexural strength test. The arrow points indicate crack line.

All the experimental groups showed lower average flexural strength compared to the control group. However, the flexural strength was found to be ≥ 500 MPa in all the specimens, which is clinically permissible.[13] Group C showed lower flexural strength then other experimental groups and Control group (Table 3). Fig. 5 shows the representative FE-SEM images of the fractured surface, where crack lines spreading in a wave form from the origin of the fracture were observed.

DISCUSSION

Y-TZP zirconia is known to be transformed by phase change, strain, and flaw during grinding and polishing of the fabrication and adjustment process for prostheses.[5,7] When strain and increase in temperature leads to excessive phase change, it can lead to an increase in surface roughness, microcraters, and grain pull-out, thereby negatively affecting the strength and reliability of the zirconia prostheses.[17,18] The phase changes in zirconia that occur after grinding or polishing are typically characterized by a change in its metastable tetragonal phase to the monoclinic phase, and the decrease in mechanical properties of Y-TZP is attributed to this type of excessive phase change.[1,5]

In this study, the phase changes in zirconia were assessed and analyzed using XRD. In this method, the specimens are irradiated with X-ray, and the diffraction angles are measured in order to determine their crystal structure. It is difficult to determine the absolute crystal structures; however, the relative crystal structure can be easily determined based on previously obtained data and is typically used to observe phase change. In the XRD analysis of zirconia, the representative tetragonal and monoclinic phase peaks are usually observed at 28 - 32 θ.[19]

A previous study on the phase changes in zirconia reported an absence of the peak for monoclinic phase after grinding, and instead report a phase change to the rhombohedral phase.[6] However, in this study, a 100% tetragonal phase distribution was observed in the control group, while in the experimental groups, a clear and reproducible monoclinic phase peak was observed, thereby indicating a phase change to the monoclinic phase. This result is similar with previous articles which reported monoclinic phase after surface treatment of zirconia.[11,12]

The relative amount of monoclinic phase (X_m) was small in all experimental groups. These results are consistent with the previous studies indicating that grinding does not significantly contribute to the phase change from the tetragonal to monoclinic phase.[5,8] However, the relative amount of monoclinic phase (X_m), TZD and H (Δ) values were found to be higher statistically in Groups B and A, compared to Group C and the control group.

In general, the heat produced during grinding and stress contributes to the phase change, and the stress is usually affected by the type of grinding apparatus, grit size, motor speed, and the force exerted during grinding or polishing.[2] In other words, many complex variables can affect the phase changes.

In this study, dedicated zirconia-specific grinding burs were used, as recommended by the manufacturer, for Groups A and B. The advantages of these zirconia grinding burs include the high grinding efficiency and decrease in heat generation. For Group C ground with Dura-Green bur which is not for zirconia grinding, the grinding time took long owing to low grinding efficiency of bur and heat can be generated, but specimens exhibited low phase changes. These results do not agree well with the results of prior studies, indicating that at 100 - 400°C, the rate of phase

change to the monoclinic phase increases.[2,4] However, Kosmac *et al.*[8] reported that when the temperature was increased to 350°C or above, a significant decrease in the fraction of monoclinic phase was observed. Swain and Hannink[20] reported that hand grinding had a five-fold higher monoclinic phase fraction than machine grinding. As a result of their study, hand grinding is more effective in inducing t→m phase transformation. On the other hand, machine grinding induces increasing the local temperature, and it can cause the reverse m→t phase transformation. Other studies also reported a decrease in the monoclinic phase due to reverse phase transformation by the excessive grinding and heat generation.[21,22] Therefore, even though the fraction of phase change depending on the increase in temperature is still debated, we believe that the local temperature increase due to excessive grinding and sparks may induce a reverse phase change to the tetragonal phase, which is more stable at high temperature.[7,13]

Surface processing such as grinding and polishing usually leads to an increase in the roughness.[18] In this study, the roughness increased in Groups A and B compared to the control group; however, Group C did not show any statistically significant differences compared to the control group. In some studies, after grinding with a bur which has a small grit size or when the surface was sandblasted, either no changes in roughness or a decrease in roughness was observed. This may be due to the removal of the milling trace.[5]

The FE-SEM analysis revealed a regular crystal structure in the control group, while traces of the grinding bur were found in the experimental groups. Group C showed a smoother grinding surface.

Albakry *et al.*[23] reported that the flexural strength test of easily breakable ceramic material show asymmetry and improbability of test results. Kosmac *et al.*[7] reported that the size of the flaw in the origin of the crack has a strong effect on the flexural strength. Therefore, the strength of ceramic material can be measured by testing its biaxial flexural strength, and because the stress is concentrated in the center, fractures can be prevented in the marginal regions.[5]

Grinding or polishing can affect to the flexural strength of zirconia.[7,8] Several factors such as volume increase from excessive phase change, formation of cracks, and surface flaw may decrease the strength of TZP.[24]

Microcracks or flaw that arises due to surface grinding act as stress concentration sites, which magnify the applied stresses. This surface defects generally contribute to the decrease in flexural strength of zirconia.[11] In this study, in the three experimental groups where grinding was conducted, the average flexural strength was lower than that in the control group. However, there was no significant difference in flexural strength between Groups A, B and control group. It can be explained by other study[5] which mentioned that appropriate amounts of phase changes can prevent the decrease in flexural strength and even increase the strength of TZP operating through the transformation toughening mechanism.

In particular, Group C showed the lowest average flexural strength and the widest flexural strength distribution, indicating a possible decrease in the strength and reliability of the zirconia. The surface roughness in Group C was lower than Groups A and B. So, the lowest value of flexural strength is considered to be caused by the grinding time and heat generation than the roughness. Group C exhibited a 1.5 - 2 fold longer grinding time owing to the low grinding efficiency, and it is possible that exposure to stress for an extended period has resulted in an increase in the factors contributing to the formation of cracks. This result is consistent with the findings in other researches that grinding can induce residual compressive stresses that increase the flexural strength of zirconia, but severe grinding can introduce deep surface flaws that act as stress concentrators and can reduce strength value.[9,13]

In addition, the lower strength values in group C can be explained due to smaller amount of monoclinic phase than other experimental groups. But, although much higher phase change was observed in Groups A and B compared to Group C, the relative amounts of monoclinic phase in experimental groups were small between 3.25% and 5.99%. So, it is questionable whether the small difference in amount of phase change between groups has influence in flexural strength and how much amount of phase change is appropriate for increasing flexural strength of zirconia. However, all experimental groups, where grinding was conducted, had more than 500 MPa flexural strength which is a value that exceeds the occlusal loads commonly recorded intraorally.[13] The strength values in this study were similar with other researches conducted regarding influence of surface treatments on the flexural strength of zirconia.[12,13]

The limitation of this study is that various factors may affect the flexural strength, and hence, it may be difficult to assess the strength and reliability solely based on the average flexural strength. In these cases, it may be easy to assess the strength by using Weibull distribution,[23] and further research is needed to test this assumption.[5,7] In addition, further studies for the phase change, heat generation and flexural strength using high speed and low speed burs which have different grit sizes would have to be implemented.

CONCLUSION

The results of this study show that the use of dedicated zirconia-specific grinding burs such as Dura-Green DIA and AllCeramic SuperMax resulted in a decrease in the grinding time and did not significantly affect the flexural strength of zirconia, and therefore, they may be recommended. However, dedicated zirconia-specific grinding burs include diamond particles to increase grinding efficiency, which in turn makes rough surface after grinding. Therefore, a fine polishing process, as recommended by the manufacturer, should be accompanied.

Surface treatment of zirconia prosthesis, such as grinding and polishing, may lead to a decrease in their strength and reliability. Therefore, efforts should be made to mini-

mize the adjustments through accurate clinical and laboratory procedures for dental prostheses because surface grinding such as occlusal adjustment may decrease the strength and longevity of zirconia based prostheses.

ORCID

Kyung-Rok Lee *http://orcid.org/0000-0002-4826-9821*
Han-Cheol Choe *http://orcid.org/0000-0003-1966-781X*
Yu-Ri Heo *http://orcid.org/0000-0002-1806-3822*
Jang-Jae Lee *http://orcid.org/0000-0001-8787-9940*
Mee-Kyoung Son *http://orcid.org/0000-0001-9225-1744*

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