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Comparison of surface topography and roughness in different yttrium oxide compositions of dental zirconia after grinding and polishing

Hyun-Sub Shin, Joon-Seok Lee*
Department of Prosthodontics, College of Dentistry, Dankook University, Cheonan, Republic of Korea

ORCID
Hyun-Sub Shin
https://orcid.org/0000-0002-4206-6652
Joon-Seok Lee
https://orcid.org/0000-0003-2962-1380

PURPOSE. The purpose of this study was to compare the surface roughness, phase transformation, and surface topography of dental zirconia with three different yttrium oxide compositions under same grinding and polishing conditions. **MATERIALS AND METHODS.** Three zirconia disks (IPS e.max ZirCAD LT, MT, MT multi, Ivoclar Vivadent AG, Schaan, Liechtenstein) were selected for experimental materials. Sixty-nine bar-shaped specimens were fabricated as 12.0 × 6.0 × 4.0 mm using a milling machine and glazing was conducted on 12.0 × 6.0 mm surface by same operator. With a custom polishing device, 12.0 × 6.0 mm surfaces were polished under same condition. Surface roughness (Ra[μm]) was measured before grinding (C), after grinding (G), and at every 3 steps of polishing (P1, P2, P3). X-ray diffraction and FE-SEM observation was conducted before grinding, after grinding, and after fine polishing (P3). Statistical analysis of surface roughness was performed using Kruskal-Wallis test and Mann-Whitney-U test was used as a post hoc test (α = .05). **RESULTS.** There were no significant differences of surface roughness between LT, MT, and MM groups. In LT, MT, and MM groups, P3 groups showed significantly lower surface roughness than C groups. X-ray diffraction showed grinding and polishing didn't lead to phase transformation on zirconia surface. In FE-SEM images, growths in grain size of zirconia were observed as yttrium oxide composition increases. **CONCLUSION.** Polished zirconia surface showed clinically acceptable surface roughness, but difference in yttrium oxide composition had no significant influence on the surface roughness. Therefore, in clinical situation, zirconia polishing burs could be used regardless of yttrium oxide composition. [J Adv Prosthodont 2021;13:258-67]

KEYWORDS
Zirconia; Dental polishing; Composition; Roughness; X-ray diffraction

Corresponding author
Joon Seok Lee
Department of Prosthodontics, College of Dentistry, Dankook University, Dankook Dentual Hospital, 119 Dandae-ro, Dongnam-gu, Cheonan 31116, Republic of Korea
Tel +82415500253
E-mail joon322@dankook.ac.kr

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INTRODUCTION

Recently, cosmetic dentistry encountered changes with advancement of dental zirconia and CAD-CAM (computer aided design-computer aided manufacturing) technique.[1] For the demand of esthetic and mechanical properties, zirconia has been widely used in dentistry. Pure zirconia exists on 3 phases (monoclinic, tetragonal, and cubic phase) under different temperatures and pressures. In phase transformation from tetragonal to monoclinic phase, 3 to 5% of volume increase occurs and leads to high mechanical properties of zirconia.[2-4] This phenomenon is called transformation toughening, and by the method of adding yttrium oxide (Y_2O_3), calcium oxide (CaO), magnesium oxide (MgO) and other additives, zirconia could be stabilized in tetragonal phase at room temperature.[1-4]

The first commercialized yttrium-stabilized zirconia(Y-TZP) had 3 mol% yttrium oxide composition (3Y-TZP) and was used in posterior regions for its high compressive stress, but it was not recommended in anterior regions requiring esthetics for its opaque shades.[5] To improve its limitation, more translucent zirconia such as 4Y-TZP and 5Y-TZP have been developed.

Translucency of zirconia can be improved by increasing yttrium oxide composition and its grain size.[6] As yttrium oxide composition increases, the amount of isotropic cubic phase increases at grain boundaries, which can cause birefringence decrease.[7-9] Also, translucency can be improved by increasing grain size that leads to decrease of grain boundaries.[2,10,11] However, increasing yttrium oxide composition can lead to decline of surface tetragonal phase composition, which has potential of surface transformation toughening; as a result, mechanical properties of zirconia would be impaired.[12-14]

Prostheses made of zirconia frequently need grinding process to change the contour of restoration and adjust the occlusion. By uncontrolled pressure and temperatures in adjustment process, excessive phase transformation and volume increase may lead to generation of micro-cracks and surface flaws and as a result, increase of surface roughness may occur.[1,2,4,15] Several studies were conducted on the wear of antagonist in contact with rough surfaces of zirconia. Janavula and Mitov *et al*. reported that rough surfaces of zirconia induced the wear of opposing enamels, and finely polished surfaces showed similar or lower surface roughness than glazed one.[16,17] From these results, zirconia polishing was proved to be of great importance to prevent the wear of antagonist. Although, difference of yttrium oxide compositions could induce different surface characteristics such as various grain sizes and tetragonal/monoclinic phase proportions.[2,4] However, in clinical situations, zirconia polishing burs are applied in 3Y-TZP, 4Y-TZP, and 5Y-TZP without considering different surface characteristics in the same way.

Therefore, the aim of this study was to compare the surface roughness of zirconia with different yttrium oxide compositions under same grinding and polishing conditions. A null hypothesis was that yttrium oxide composition would have no effect on the difference of surface roughness under same polishing conditions.

MATERIALS AND METHODS

69 specimens (n = 23 per group) were fabricated using zirconia disks with three different yttrium oxide compositions (Table 1). 60 specimens were used for surface roughness measurement and X-ray diffraction after sequential polishing procedures and 9 were used for FE-SEM observation. Rectangular-shaped specimens (12.5 × 6.5 × 4.5 mm) were designed using CAD program (Solidworks, Dassault Systèmes, Paris, France) and fabricated by a milling machine (Cameleon CS, Neo Biotech Co., Seoul, Korea). Especially, as MT multi disks were multi-layered so that dentin zone was 4Y-TZP and incisal zone was 5Y-TZP, specimens of MT multi disks were designed as one 12.5 × 6.5 mm surface being located in incisal zone, which was 5Y-TZP (Fig. 1). Then, specimens were sintered at 1500°C according to the manufacturer's recommendation. After precise machining, final dimension of specimens was set as 12.0 × 6.0 × 4.0 mm. One 12.0 × 6.0 mm surfaces planned to be experimented were wet-polished with 800-, 1000- and 1200- grit sandpapers (HD-PM-100, Hando, Busan, Korea) and glazed with layering technique (IPS Ivocolor Glaze Paste, Ivoclar Vivadent AG, Schann, Liechtenstein) by single

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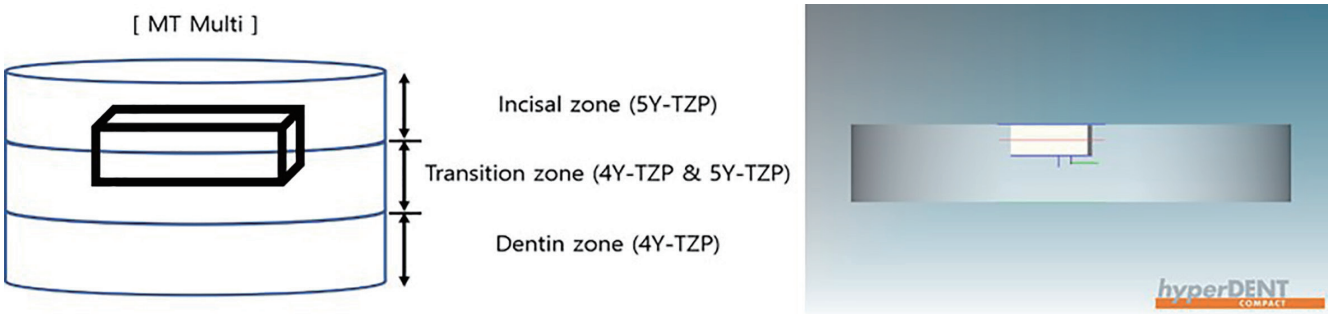

Fig. 1. Manufacturing methods of specimens in MM groups.

Table 1. Manufacturer's specifications and chemical compositions of selected materials

Material	Manufacturer	Composition
IPS e.max ZirCAD LT	Ivoclar Vivadent AG (Schaan, Liechtenstein)	Zirconium oxide (≤ 88.0 - 95.5 wt%) Yttrium oxide (4.5 - 6.0 wt%) Hafnium oxide (≤ 5.0 wt %) Aluminium oxide (≤ 1.0 wt%) Other oxides (≤ 1.0 wt%)
IPS e.max ZirCAD MT	Ivoclar Vivadent AG (Schaan, Liechtenstein)	Zirconium oxide (≤ 86.0 - 93.5 wt%) Yttrium oxide (6.5 - 8.0 wt%) Hafnium oxide (≤ 5.0 wt %) Aluminium oxide (≤ 1.0 wt%) Other oxides (≤ 1.0 wt%)
IPS e.max ZirCAD MT multi	Ivoclar Vivadent AG (Schaan, Liechtenstein)	Zirconium oxide (≤ 86.0 - 93.5 wt%) Yttrium oxide (6.5 - 8.0 wt%) Hafnium oxide (≤ 5.0 wt %) Aluminium oxide (≤ 1.0 wt%) Other oxides (≤ 1.0 wt%)

Table 2. Classification and abbreviations of test groups

Materials	Surface conditions				
	Control	Grinding	Coarse polishing	Medium polishing	Fine polishing
IPS e.max ZirCAD LT (LT)	LT-C	LT-G	LT-P1	LT-P2	LT-P3
IPS e.max ZirCAD MT (MT)	MT-C	MT-G	MT-P1	MT-P2	MT-P3
IPS e.max ZirCAD MT multi (MM)	MM-C	MM-G	MM-P1	MM-P2	MM-P3

operator. Specimens with only glazing were set as a control group, and experimental groups underwent sequential grinding and polishing procedures. Classification and abbreviation of test groups were described at Table 2.

To perform surface treatments in the same condition, a custom device was fabricated (Fig. 2). A low-speed handpiece was installed in the device and cylinder-shaped zirconia grinding burs (Pre-Zirpol Dia HP, Neobiotech Co., Seoul, Korea) and 3-step polishing burs (Zirpol Dia HP, Neobiotech Co., Seoul, Korea) were applied (Fig. 3). 12.0 × 6.0 mm surfaces in each specimen were processed under 2 N pressure and 15,000 rpm according to the manufacturer's recommendation. Also, 0.1 mm/s speed of 10 swiping motion was performed in each step with air-cooling. In every step, specimens were cleaned with ultrasonic cleaning device for 10 minutes and dried for 24 hours

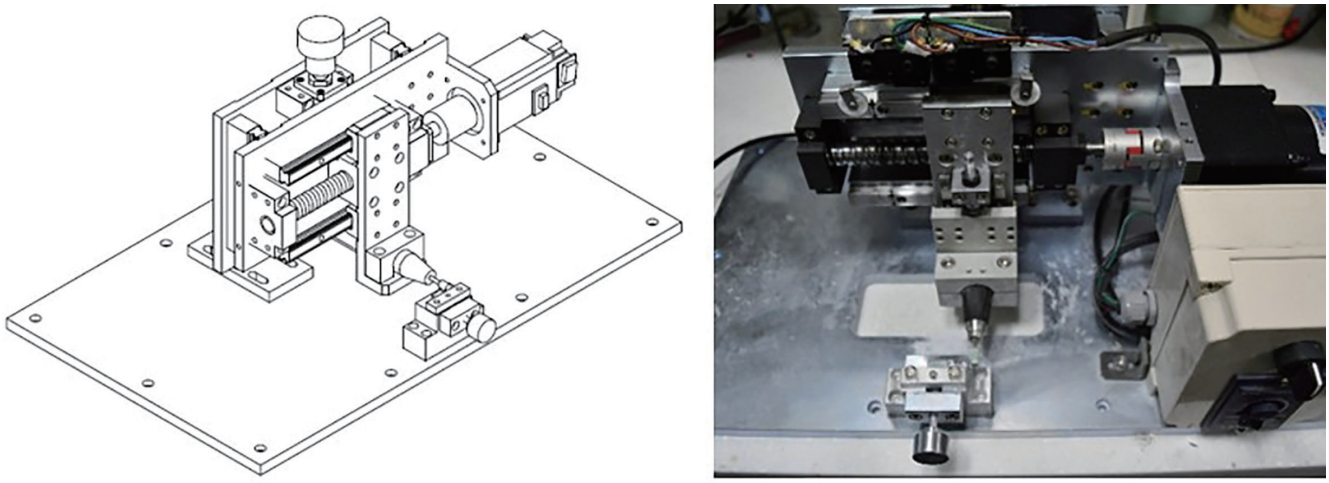

Fig. 2. Custom device for grinding and polishing.

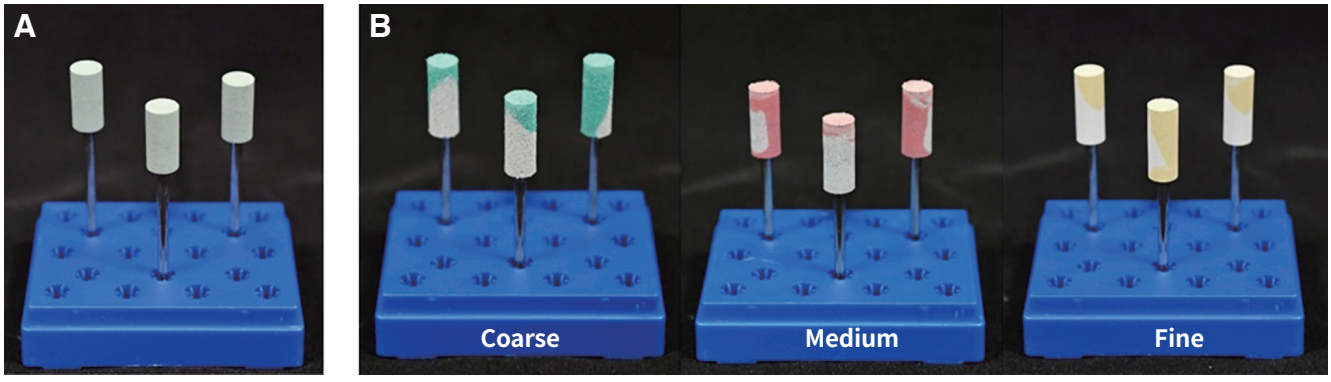

Fig. 3. Zirconia grinding and polishing burs. (A) Pre-zirpol Dia HP, (B) Zirpol Dia HP.

at room temperature.

Surface roughness(Ra[μm]) was measured in control group and every grinding and polishing steps used contact surface profiler (Tencor D-300, KLA-Tencor Co., Milpitas, CA, USA). In every step, 3 measurements were performed parallel to grinding and polishing direction and the average surface roughness was calculated. Measurement conditions were 3.0 mm distance, 0.1 mm/s using 2.0 μm diameter probing tip with 15.0 mg pressure.

X-ray diffraction was performed for analyzing phase transformation of specimen surfaces in control groups, grinding groups, and fine polishing groups. A specimen in each group was randomly selected and X-ray diffraction was conducted using X-ray diffractometer (Smartlab, Rigaku Co., Tokyo, Japan). Experimental conditions were 40 kV, 30 mA, 10° to 70° 2θ, 0.02°/step size.

Surface structures were analyzed using a field emission scanning electron microscope (FE-SEM) (Apero S HiVac, FEI Company, Hillsboro, OR, USA) in control groups, grinding groups, and fine polishing groups. A specimen in each group was randomly selected and underwent thermal etching in 1,300°C (10°C/min) for 20 minutes. Surface observations were performed at 30000× magnification.

The statistical analysis was conducted using IBM SPSS statistics v27.0 (IBM Corp., Chicago, IL, USA). Kruskal-Wallis test was performed to assess the effect of yttrium oxide compositions on surface roughness. Mann-whitney-U-test was used as a post hoc test and statistical significance was set at P <.05.

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RESULTS

Table 3 shows the mean and standard deviation of surface roughness. In all zirconia groups, surface roughness according to sequential grinding and polishing and surface roughness in all zirconia groups tended to decrease and P3 showed the lowest surface roughness than other states. In control group, MT showed the highest surface roughness (0.19 ± 0.05 μm). Compared in different yttrium oxide compositions, in P2 which indicates medium polishing state, there were significant difference between LT, MM groups ($P < .001$) and MT, MM groups ($P < .001$) (Table 3). When comparing surface roughness in single zirconia group, P2 in all zirconia groups showed similar surface roughness with control groups. On the other hand, P3 which was fine polishing state showed significantly lower surface roughness than control groups in three zirconia groups ($P < .05$) (Table 3).

The XRD patterns for three zirconia groups are presented in Fig. 4. XRD patterns for each zirconia group in different surface conditions were compared. In XRD results, all groups showed similar patterns of main peaks and minor peaks that represents tetragonal phase in different surface treatments.

SEM images showed different surface characteristics of specimens in different polishing state (Fig. 5, Fig. 6, and Fig. 7). Surfaces of control groups showed even surfaces, but some grains and pores can be observed (Fig. 5). Grinded zirconia surfaces showed morphological changes and many scratches were observed (Fig. 6). In polished state, surfaces showed reduced scratches and textures compared to grinding groups (Fig. 7). As yttria oxide composition increased, sizes of zirconia grains tended to increase.

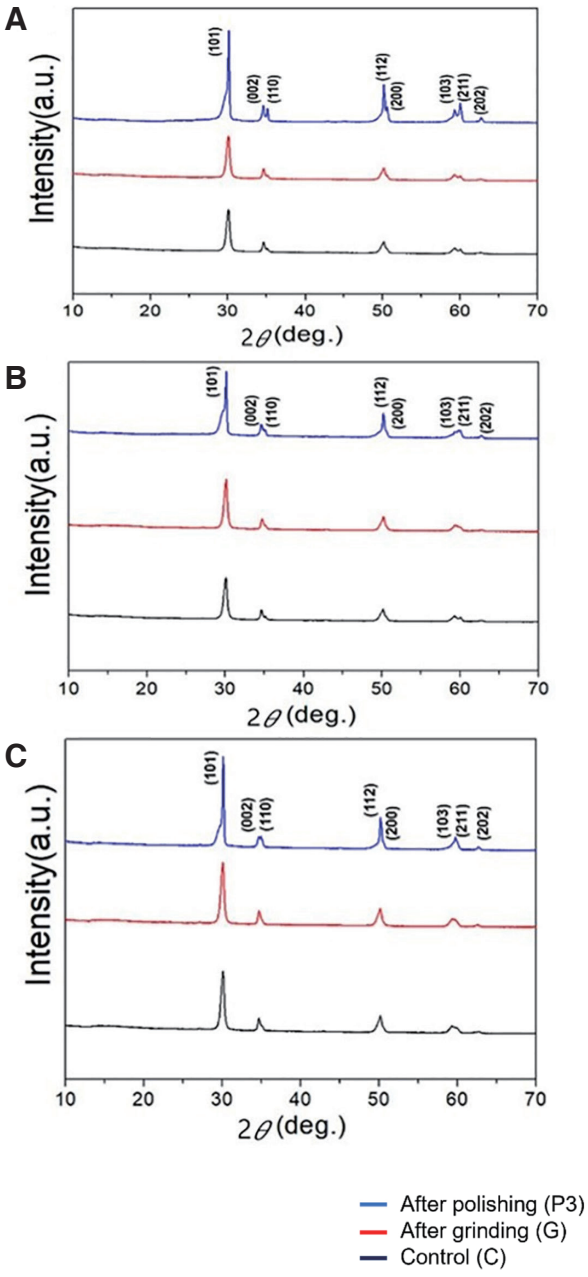

Fig. 4. XRD patterns of the surface of zirconia specimens. (A) LT, (B) MT, (C) MM.

Table 3. Surface roughness (Ra) with standard deviations of all groups

Group	Mean ± SD (μm)				
	C	G	P1	P2	P3
LT	0.14 ± 0.06 ^{a1}	0.36 ± 0.10 ^{a2}	0.26 ± 0.06 ^{a3}	0.18 ± 0.03 ^{a1}	0.07 ± 0.02 ^{a4}
MT	0.19 ± 0.05 ^{a1}	0.36 ± 0.09 ^{a2}	0.27 ± 0.07 ^{a3}	0.19 ± 0.04 ^{a1}	0.03 ± 0.03 ^{a4}
MM	0.15 ± 0.06 ^{a1}	0.33 ± 0.09 ^{a2}	0.28 ± 0.06 ^{a3}	0.13 ± 0.04 ^{b1}	0.08 ± 0.03 ^{a4}

LT: IPS e.max ZirCAD LT, MT: IPS e.max ZirCAD MT, MM: IPS e.max ZirCAD MT multi
C: control, G: grinding, P1: Coarse polishing, P2: Medium polishing, P3: Fine polishing
Different lowercase letters show significant differences among different zirconia groups ($P < .05$).
Different numbers show significant differences among different polishing states ($P < .05$).

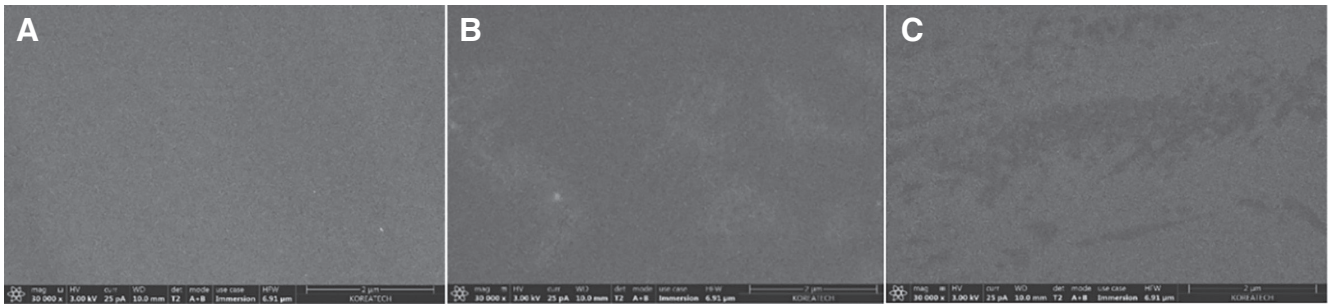

Fig. 5. Scanning electron micrographs of control group (magnification ×30000). (A) LT, (B) MT, (C) MM.

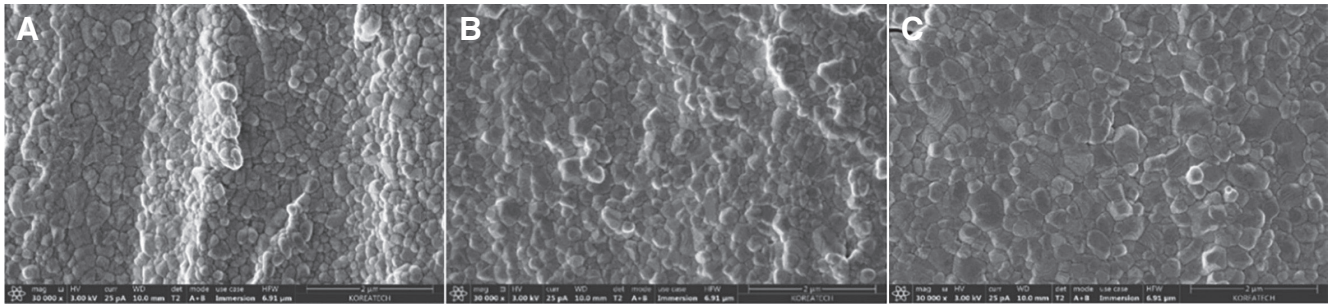

Fig. 6. Scanning electron micrographs of grinding group (magnification ×30000). (A) LT, (B) MT, (C) MM.

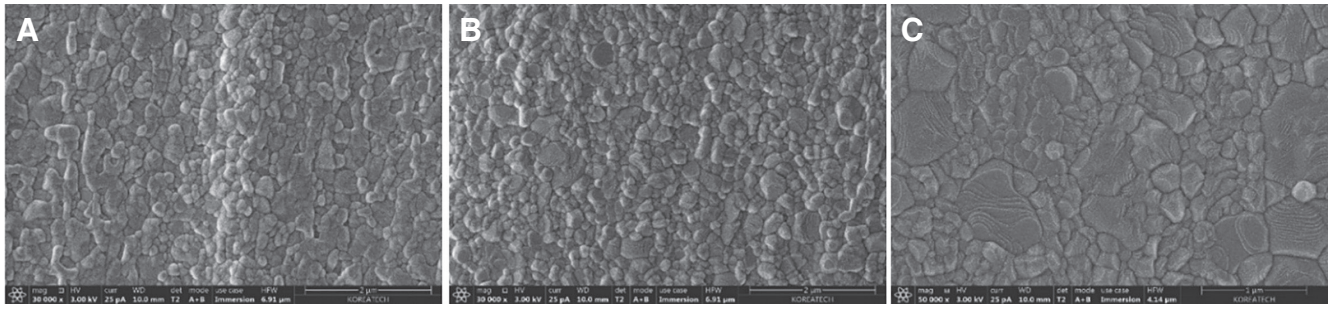

Fig. 7. Scanning electron micrographs of fine-polishing group (magnification ×30000). (A) LT, (B) MT, (C) MM.

DISCUSSION

The aim of this study was to compare the surface roughness between dental zirconia with different yttrium oxide compositions under same polishing condition. Many previous studies about surface roughness of zirconia had limitations that they lacked comparison between various yttria compositions or conditions simulating clinical situations. Most studies on surface roughness and effectiveness of polishing systems on zirconia was conducted with only single yttria compositions and usually 3Y-TZP were selected. Goo *et al*.[18] compared the surface roughness of 3Y-TZP treated with 5 polishing systems. Huh *et al*.[19] studied the effects of 6 polishing systems on surface roughness and phase transformation of 3Y-TZP. Otherwise, Khayat *et al*.[20] compared surface roughness of 5Y-TZP using 5 polishing systems. In studies comparing between various yttrium oxide compositions, Amarante *et al*.[21] examined milled surfaces of 3Y-TZP and 5Y-TZP without grinding and polishing using clinical zirconia burs. Also, Vila-Nova *et al*.[22] examined 3Y-TZP and 5Y-TZP with applying only 1-step polishing system. Although Zhang *et al*.[5] recently compared

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the surface roughness and wear-resistance of 3Y-TZP, 4Y-TZP, and 5Y-TZP, specimens were mirror-polished and grinding and polishing with clinical burs were not conducted. Like Zhang's study, few studies were conducted with comparing surface roughness of 3Y-TZP, 4Y-TZP, and 5Y-TZP after being treated with zirconia polishing burs. For the purpose of complementing previous studies, 3 types of zirconia with different yttrium oxide compositions (3Y-TZP, 4Y-TZP and 5Y-TZP) were selected and underwent grinding and polishing procedures with clinical zirconia burs.

For grinding and polishing specimens under constant condition, a custom device was fabricated. Although many studies about zirconia polishing systems had been conducted, there were no standardized polishing conditions including pressure, rpm, time, etc. Unlike polishing rpm, there were no manufacturer's recommendations about pressure, so pressure in this study was set as 2 N referring to the studies of Hmaidouch *et al*.[23] and Chavali *et al*.[24] To simulate chair-side clinical condition, specimen grinding and polishing were done with air-cooling rather than water-cooling method.

Arithmetical mean roughness (Ra) was used for representing the surface roughness of dental zirconia. Ra is widely used index in dental studies, but it has a limitation that fails to give exact information including presence of excessively protruded or undermined regions. To measure the Ra, a linear contact surface profiler was used. This device has a disadvantage compared to optical surface profiler in that it could not calculate mean surface roughness in specific area. Thus, different results might be drawn if measured by another type of devices.

In this study, as surface roughness in different yttrium oxide compositions didn't show significant difference, the null hypothesis was accepted. According to the studies of Deville *et al*.[25] and Haraguchi *et al*.,[26] zirconia under phase transformation showed the increase of surface roughness. Lucas *et al*.[27] reported that under phase transformation, grains on the zirconia surface expanded in volume pushing one another and, as a result, surface roughness could increase. From those studies, as difference of tetragonal phase composition in LT, MT, and MM groups existed, different amounts of surface roughness were expected in that phase transformation amounts may differ. However, surface roughness between LT, MT, and MM groups didn't show significant differences. This result corresponded to the study of Amarante *et al*.[21] who reported that when polishing 5Y-TZP and two types of 3Y-TZP using diamond polishing suspension, surface roughness didn't show significant difference. In the study of Zhang *et al*.[5] who examined the surface roughness of 3Y-TZP, 4Y-TZP, and 5Y-TZP under diamond polishing suspension, the author reported that there was no significant difference of surface roughness among different yttrium oxide compositions and it corresponded to the result of the current study. Chavali *et al*.[24] examined the maximum temperature when polishing zirconia for 1 minute with 2 N pressure, which was same pressure of this study, and various RPM. In same pressure and rpm set in this study without using coolants, maximum temperature was measured at 34.1°C and temperature changes of zirconia did not exceed 17°C. Also, regarding the polishing pressure, Fischer *et al*.[28] evaluated the effects of bur application force on zirconia surface topography. When compared among three different pressure (1.0 N, 4.5 N, and 11 N), there were no significant difference among the surface roughness of all groups in atomic force micrograph (AFM) and optical profilometer. As pressure in current study was just close to the least pressure in Fischer's study, it wasn't enough to induce destructive surface damages. From Chavali and Fischer's study, it seemed that polishing conditions set in this study didn't generate pressure and temperature enough to induce phase transformation on the zirconia surface. This could be observed in XRD results and they will be discussed later in this section.

Compared in single yttrium oxide composition, fine polishing groups (P3) showed lower surface roughness than glazing groups (C) ($P < .05$). It has been reported that finely polished zirconia has lower surface roughness than glazed zirconia using layering technique and it corresponded to the result of this study.[29,30] Otherwise, Preis *et al*.[31] reported similar surface roughness between polished zirconia and glazed one. Glazing was done using layering technique that mixes glazing powders and liquids. Layering technique is a manual laboratory work. Thus, inappropriate powder-liquid mix, uneven layer thick-

ness and other errors may occur, and it could induce rough zirconia surfaces.[32] Surface roughness under medium polishing state was about 0.2 μm in all groups (LT: 0.18 ± 0.03 μm. MT: 0.19 ± 0.04 μm, MM: 0.13 ± 0.04 μm). Also, there was no significant difference between surface roughness of glazing groups (C) and medium polishing groups (P2). Mai *et al*.[33] evaluated the effects of 4 protocols of zirconia finishing and polishing on surface topography and bacterial adhesion of zirconia surfaces. Protocols were as follows: coarse finishing alone, coarse finishing and medium polishing, coarse finishing and fine polishing, and coarse finishing, medium polishing, and fine polishing. Authors suggested that coarse-grit finishing and medium-grit polishing were essential steps in that smoothing grooves and scratches could be achieved by this sequential protocol. In case of intraoral restorations, surface roughness lower than 0.2 μm (Ra) is ideal for prevention of plaque accumulation.[34,35] Also, Jones *et al*.[36] reported that surface roughness of dental restoration under 0.5 μm (Ra) could not be detected by tongue sensation. Referring to values from studies about minimal surface roughness of dental restoration, surface roughness in medium polishing state of zirconia would be clinically acceptable.

X-ray diffraction was conducted in control groups (C), grinded groups (G), and fine polishing groups (P3). In XRD analysis, all groups showed similar patterns of main peaks and minor peaks that represents tetragonal phase in every steps. Also, change of peaks that represent monoclinic phase could not be observed. These results corresponded to other studies that zirconia polishing didn't induce phase transformation.[20,37,38] However, Souza *et al*.[39] reported that when grinding and polishing 3Y-TZP, monoclinic transformation could be observed and this result seemed to come from different experimental conditions such as pressure, rpm, and other factors.

In FE-SEM observation, morphological changes as to grinding and polishing were observed in all groups. As yttrium oxide composition increased, larger grain size was observed. Recently, there were challenging reports about phase composition of dental zirconia. Harada *et al*.[40] observed phase composition of 5Y-TZP before and after low-temperature degradation. Using backscattered electron image (BSE), 5Y-TZP surfaces were divided into L-region (Ø > 1 μm) and S-region (Ø < 1 μm) by grain sizes. Then, energy dispersive X-ray spectroscopy (EDX) was conducted on these regions and the results showed that L-region with large grain size has significantly higher yttrium oxide compositions than S-legion (P < .01). Also, they analyzed that pseudo-cubic phase existed on L-region by using XRD. Gibson and Irvine[41] reported that under 8 mol% yttrium oxide composition, dental zirconia was composed of pseudo-cubic phase rather than cubic phase. Although these reports should be carefully discussed and further studies should be conducted, results of these reports might give the information explaining the indifferent surface roughness of various zirconia in this study.

There are some limitations in this study. Disks of zirconia and zirconia burs from only a single company were used and specimens didn't undergo aging. Also, polishing conditions including pressure, rpm, time, cooling methods, and other factors could be changed, and it could affect the results. Further studies should be conducted using additional products under various conditions.

CONCLUSION

From this study, following conclusions could be drawn. Zirconia with different yttrium oxide compositions showed similar surface roughness after polished with zirconia polishing burs. Finely polished zirconia showed better surface roughness than glazed one. In 3-step zirconia polishing kit, zirconia polished with up to medium polishing burs showed clinically acceptable surface roughness. Thus, zirconia polishing burs in this study could be applied to zirconia with three different yttrium oxide compositions (3Y-TZP, 4Y-TZP, and 5Y-TZP) in the same way.

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