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Analysis of surface characteristics of (Y, Nb)-TZP after finishing and polishing

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PURPOSE. This *in vitro* study aimed to evaluate the surface characteristics of a full veneer crown fabricated chairside (CS) from a (Y, Nb)-TZP zirconia block in response to conventional zirconia grinding and polishing. **MATERIALS AND METHODS.** Zirconia crowns (n = 40) were first prepared and divided into two groups of materials: Labside (LS) and CS, after which each specimen went through a five-step grinding and polishing procedure. Following each surface treatment, surface characteristics were analyzed using confocal laser microscopy (CLSM), average surface roughness (Ra) values were processed from the profile data through Gaussian filtering, and X-ray diffraction pattern analysis was performed to evaluate the monoclinic (M) phase content. Then, a representative specimen was selected for field-emission scanning electron microscopy (FE-SEM), followed by a final analysis of the roughness and X-ray diffraction of the specimens using the independent t-test and repeated measures analysis of variance (RM-ANOVA). **RESULTS.** In every group, polishing significantly reduced the Ra values ($P < .001$). There was no significant difference in Ra between the polished state CS and LS. Furthermore, CLSM and FE-SEM investigations revealed that even though grain exposure was visible in CS specimens throughout the as-delivered and ground states, the exposure was reduced after polishing. Moreover, while no phase transformation was visible in the LS, phase transformation was visible in CS after every surface treatment, with the M phase content of the CS group showing a significant reduction after polishing ($P < .001$). **CONCLUSION.** Within the limits of this study, clinically acceptable level of surface finishing of (Y, Nb)-TZP can be achieved after conventional zirconia polishing sequence. [J Adv Prosthodont 2022;14:335-45]

KEYWORDS
Fully sintered zirconia; Partially sintered zirconia; CAD-CAM restoration; Surface roughness; Phase transformation

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INTRODUCTION

The chairside (CS) CAD-CAM system needs to have a milling material that fulfills the rather contradictory mechanical characteristics of not only being mechanically strong but also having high machinability.[1,2] This requirement has increased the need for a durable material, leading to the development of pre-crystallized blocks.[3] Today, when it comes to CS restoration material selection, particle-filled glasses, specifically lithium disilicate, are therefore considered as the choice-to-go by many clinicians, with clinical studies showing promising survival rates of up to ten years.[4] However, most of these particle-filled glasses show reduced mechanical properties compared to metal or zirconia materials.[5,6] In addition, when used in high-stress bearing areas, complications, such as chipping and increased surface roughness are commonly observed.[7,8] Thus, some attempts have been made to utilize zirconia, which is a much stronger ceramic, with better surface roughness properties.[9]

Currently, most zirconia materials come in the partially sintered form, which has many advantages, one of which is its superior machinability compared to the fully sintered counterparts. However, they must be machined to be roughly 25% larger to compensate for the densification caused by sintering,[10] which requires complex calculations to account for these volumetric changes.[9,11] Besides, the conventional zirconia sintering process requires at least 4 - 7 hours to process, rendering it unfeasible for the chairside restoration.[11,12] Therefore, speed sintering schedules attempt to address these issues. Still, although most characteristics of the speed-sintered zirconia show similar results to conventional sintering, mechanical reliability and other related effects on the zirconia material are yet to be confirmed.[9,13] Moreover, even in the speed sintering mode, the sequence requires a total sintering time of 60 min,[9] increasing the CS waiting time for the patients. Thus, with the exception of a few commercially available partially sintered blocks with speed sintering sequences, majority of zirconia restorations are fabricated in labside (LS) modality.

Utilizing a fully sintered zirconia block could evade much of the issue caused by sintering. However, it has been neglected by clinicians because of its low machinability and additional expenses caused by the extreme wear of the milling bur.[14] Therefore, a fully sintered niobium oxide containing an yttria-stabilized tetragonal zirconia polycrystal ([Y, Nb]-TZP) block (Perfit-FS; Vatech MCIS, Hwaseong, Korea) was developed to address this issue. According to the manufacturer, the block presented a lower surface hardness (8.5 GPa) than the conventional fully sintered Y_2O_3-stabilized zirconia blocks,[15] which greatly improved its machinability. The flexural strength of the block was 500 MPa, capable of three-unit prosthesis involving molar restoration (ISO 6872:2015). Moreover, the block shows high resistance to low-temperature degradation and favorable fracture resistance,[15,16] with block restorations showing successful clinical performances within a six-month follow-up.[15] Still, this material lacks long-term evaluation, and certain clinical and mechanical aspects are yet to be ventured.

Surface roughness is one of the key mechanical features of zirconia and ceramic restorations. To this end, studies have reported that a dental prosthesis with increased surface roughness promotes antagonistic tooth wear, leading to many clinical problems.[9,17-19] It was discovered that rough surfaces promote dental plaque accumulation, leading to aberrant biologic responses.[20] Studies also investigated various conditions for zirconia restoration grinding and polishing.[18,19,21-25] With the information given from these studies, it is now generally accepted by clinicians to first, adjust the zirconia crowns with diamond bur connected to a high-speed handpiece under a water coolant, then to polish with a zirconia polisher connected to a low speed handpiece. Hence, many studies about polishing systems for different zirconia restorations remarkably exist. However, the ideal surface polishing conditions for the aforementioned (Y, Nb)-TZP block have not been documented. Therefore, we undertook this study to evaluate and compare the effect of different grinding and polishing methods on the surface roughness and phase transformation of (Y, Nb)-TZP. The hypothesis of this study was that surface roughness and phase transformation of a polished CS (Y, Nb)-TZP block would show significant difference from that of the conventional LS monolithic 3Y-TZP.

MATERIALS AND METHODS

Figure 1 schematically represents the study flow, with a standardized prepped acrylic-resin typodont tooth model (D85DP-500B.1; Nissin Dental, Kyoto, Japan) of the maxillary left central incisor being used for this study. First, the prepped resin teeth were adapted to their related dentiform, after which they were scanned using a laboratory scanner with an accuracy of seven microns (T700; Medit, Seoul, Korea). Then, a full-contour monolithic anterior crown was designed on the prepared tooth using dental CAD software (3Shape Dental System; 3Shape, Copenhagen, Denmark), followed by a design of the incisal third of the specimen to represent a flat surface for use in confocal laser microscopy (CLSM) and X-ray diffraction (XRD). The resulting design files were used in the subsequent milling.

This study used 40 zirconia crowns, of which 20 were fabricated for each testing group: CS and LS, according to their fabrication method. While the CS group was milled from commercially available (Y, Nb)-TZP block (Perfit-FS; Vatech MCIS, Hwaseong, Korea), using a four-axis milling machine (CoriTEC one; imes-icore, Eiterfeld, Germany), the LS group was milled from a commercially available LS 3Y-TZP disk (Perfit-HT; Vatech MCIS, Hwaseong, Korea), using a five-axis milling machine (DEG-5X100; Arum, Doowon ID, Daejeon, Korea). The LS specimens went through post-milling sintering process, as advised by its manufacturer. This state is named as as-delivered (AD) state.

Sequential grinding and polishing were conducted on each specimen. Table 1 shows the specifications for each grinding step and the burs used in the study. First, the palatal incisal third was selected for grinding and polishing, with the corresponding regions of each specimen marked with a permanent marker to ensure that all required surfaces were treated. Then, grinding and polishing were performed by a single operator with loupe inspection, similar to previous studies.[26-28] Before each step, a tactile force of 0.98 N was calibrated into an electronic scale to simulate finger pressure.[28,29] However, to standardize the grinding and polishing movement, each stroke was performed in the same mesiodistal direction, perpendicular to the teeth axis. After each step, including the AD step, all specimens underwent surface evaluation with CLSM and XRD, after which a representative specimen from the group was selected from each step for field-elec-

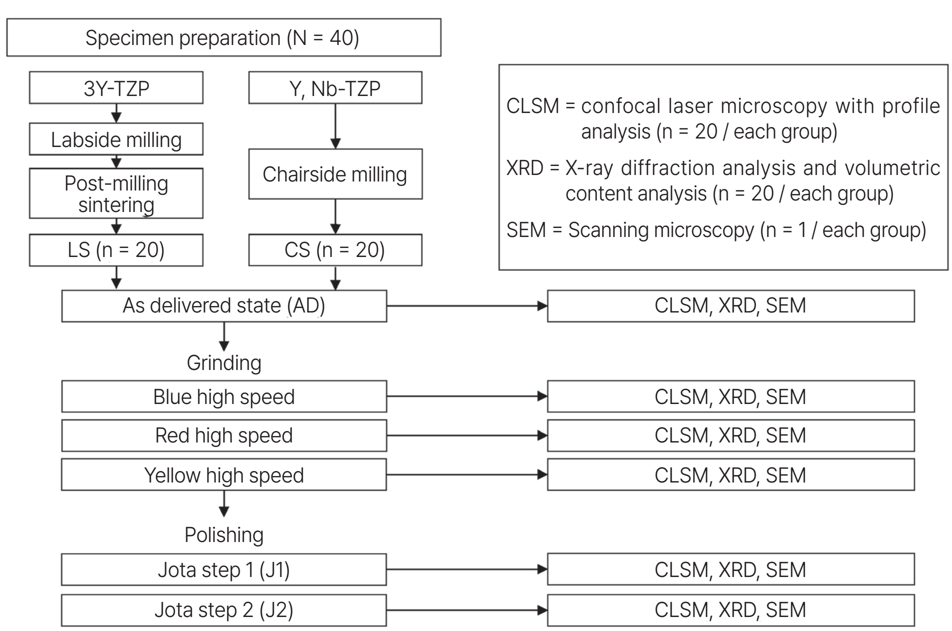

Fig. 1. Flow chart of research.
LS, labside; CS, chairside; CLSM, Confocal laser scanning microcopy; XRD, X-ray diffraction analysis; SEM, Scanning electron microscope.

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Table 1. Parameters used in this study for grinding and polishing

Procedures	Rotary specifications	Time	Directions
Grinding with blue diamond bur Standard grit (106 - 125 µm) diamond bur (blue) Dia-Burs (TR-13, Mani, Inc., Tochigi, Japan)	High speed	30 s	Mesiodistal
Grinding with red diamond bur Fine grit (53 - 63 µm) diamond bur (red) Dia-Burs (TR-13F, Mani, Inc., Tochigi, Japan)	High speed	30 s	Mesiodistal
Grinding with yellow diamond bur Extra fine grit (20 - 30 µm) diamond bur (yellow) Dia-Burs (TR-13EF, Mani, Inc., Tochigi, Japan)	High speed	30 s	Mesiodistal
Polishing with Jota kit step 1 bur Dark green, Swivel shape: medium grit size (J1) ZIR9868 (Jota kit 1434, Jota, Rüthi, Switzerland)	Low speed	60 s	Mesiodistal
Polishing with Jota kit step 2 bur Light green, Swivel shape: fine grit size (J2) ZIR9868F (Jota kit 1434, Jota, Rüthi, Switzerland)	Low speed	60 s	Mesiodistal

High-speed: A Ti-Max X 600 L NSK handpiece (NSK, Nakanishi, Japan) mounted on a high-speed coupling phatelus (PTL-CL-4HV-T; NSK, Nakanishi, Japan). rpm: 380,000-440,000
Low speed: A EX-VI E-type straight handpiece (NSK, Nakanishi, Japan) mounted on a slow-speed air motor (EX 203; NSK, Nakanishi, Japan) at 22,000 rpm

tron scanning electron microscopy (FE-SEM).

Average surface roughness (Ra) and surface topography were analyzed using a 3D confocal laser microscope (LSM 800, Carl Zeiss, Jena, Germany). The resulting 3D surface areas were processed using templates in ConfoMap software (Zeiss, Jena, Germany). Then, Ra values were derived according to the ISO 4287 parameter, using a Gaussian filter of 25 µm, with the Ra values of each specimen representing the mean ± SD from three independent scans at three different locations: the middle and 1 mm right and left from the middle. Specimens were also analyzed with an X-ray diffractometer (Bruker D8 Advance, Bruker, Karlsruhe, Germany). Data were collected at angle intervals ranging between 27° and 32° with a scan speed of 1°/min. Then, volume fractions of the monoclinic phase on the surfaces (V_m) were obtained by the Garvie and Nicholson methodology, modified by Toraya *et al*.[30] For FE-SEM, the selected representative specimens were sputter-coated with palladium and observed at magnifications of × 1,000 and × 10,000. (FE–SEM; Apreo S; Thermo Fisher Scientific, Waltham, MA, USA).

All analyses were conducted using the SPSS statistical software (IBM SPSS Statistics v26; IBM Corp., Armonk, NY, USA). First, the Ra values and volume fraction of the monoclinic phase (V_m) were evaluated using repeated measures analysis of variance (RM-ANOVA) with Greenhouse-Geisser corrections, followed by post-hoc Dunnett's test. The normality was confirmed using Shapiro-Wilk's test, and the Ra values of each group after each subsequent surface treatment (AD, blue, red, yellow, J1, and J2) were compared with independent t-test. $P < .05$ was considered statistically significant. A power analysis was performed according to previous articles, with the effect size = 2, beta error = 0.80, and alpha error = 0.95. From this calculation, specimen sizes were set to 20.[23]

RESULTS

Table 2 presents the Ra values of each group after every surface treatment. For Ra values, repeated measures ANOVA showed a significant difference after each surface treatment (F = 795.22, df = 3.56, $P < .001$, Greenhouse-Geisser corrected). Pairwise comparisons also showed that all surface treatments led to significant changes ($P < .05$), confirming our results. The profiles of the estimated marginal means are shown in Figure 2.

On the other hand, the bar graph of the Ra values for each group after every surface treatment are represented in Figure 3. Investigations revealed significant differences between the groups throughout the AD

Table 2. Mean Ra values after each surface treatment

	AD	blue	red	yellow	J1	J2
LS	0.316 ± 0.016[a,1]	0.431 ± 0.036[a,2]	0.375 ± 0.027[a,3]	0.309 ± 0.025[a,4]	0.192 ± 0.062[a,5]	0.104 ± 0.041[a,6]
CS	0.703 ± 0.039[b,1]	0.572 ± 0.072[b,2]	0.55 ± 0.057[b,3]	0.413 ± 0.052[b,4]	0.218 ± 0.04[a,5]	0.126 ± 0.03[a,6]

Significant differences ($P < .05$) between rows and columns are represented in numbers and alphabets, respectively.
AD, as delivered state; blue, blue diamond bur ground state; red, red diamond bur ground state; yellow, yellow diamond bur ground state; J1, Jota kit step 1 polished state; J2, Jota kit step 2 polished state; LS, labside; CS, chairside.

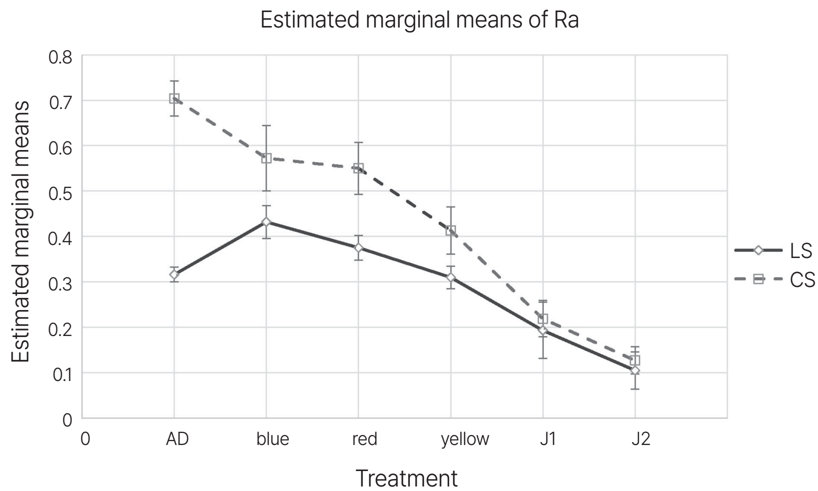

Fig. 2. Estimated marginal means profile of Ra values.
AD, as delivered state; blue, blue diamond bur ground state; red, red diamond bur ground state; yellow, yellow diamond bur ground state; J1, Jota kit step 1 polished state; J2, Jota kit step 2 polished state; LS, labside; CS, chairside.

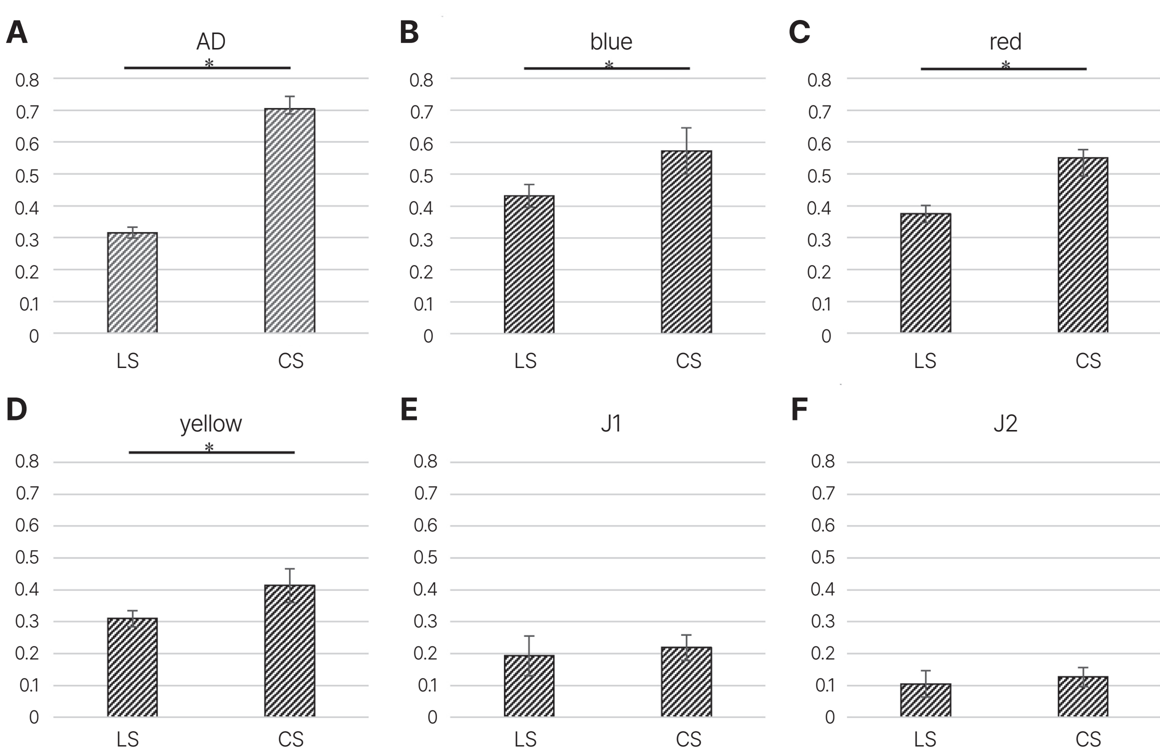

Fig. 3. Ra value comparison of each group after each state. Mean Ra values of each group are compared in each surface treatment. (A) as delivered state; (B) blue diamond bur ground state; (C) red diamond bur ground state; (D) yellow diamond bur ground state; (E) Jota kit step 1 bur polished state; (F) Jota kit step 2 bur polished state. Significant differences are annotated with asterisk ($P < .05$).
LS, labside; CS, chairside.

state and high-speed grinding. However, no significant difference existed in the polished states (J1 and J2).

The CLSM images are represented in Figure 4. Grain exposures were notably observed in the AD state CS group, including visible grinding marks throughout the grinding specimens. However, these grinding marks were markedly reduced after polishing, especially after J2.

The FE-SEM images obtained from each specimen are represented in Figure 5. Although grains of 5 - 10 µm were exposed in the CS group throughout the AD state and blue, red, and yellow ground state, these grain exposures were less visible after J1 and J2 polishing.

XRD graphs obtained during this study are represented in Figure 6. We notably observed phase transformation levels in CS but not in LS, as represented in Figure 6G. For Vm values, repeated measures ANOVA confirmed significant difference after surface treatment ($F = 282.75$, $df = 5$, $P < .001$, Sphericity assumed).

DISCUSSION

This study investigated the effect of conventional zirconia grinding and polishing on CS (Y, Nb)-TZP restoration's surface characteristics and phase transformation. The null hypothesis was partially rejected because the Ra values were significantly higher ($P < .05$) in the CS group than in the LS group throughout the AD and ground states. Moreover, compared to the 3Y-TZP that showed no phase transformation in the current experiment, CS zirconia specimens showed phase transformations in every surface treatment, including that during the AD state. However, the Ra values of the polished CS group were comparable to the LS group. Only a limited number of CS fully sintered zirconia products are currently used in dental restoration, leading to severely lacking documentation on the surface roughness characteristics of these materials. To this end, a study introduced niobium oxide into a Y-TZP material to stabilize zirconia materials from LTD-dependent phase transformation and diminish oxygen vacancies.[31-35] Paired with the fact that these materials showed superior machinability compared with the conventional fully sintered zirconia materials, it was postulated that these materials

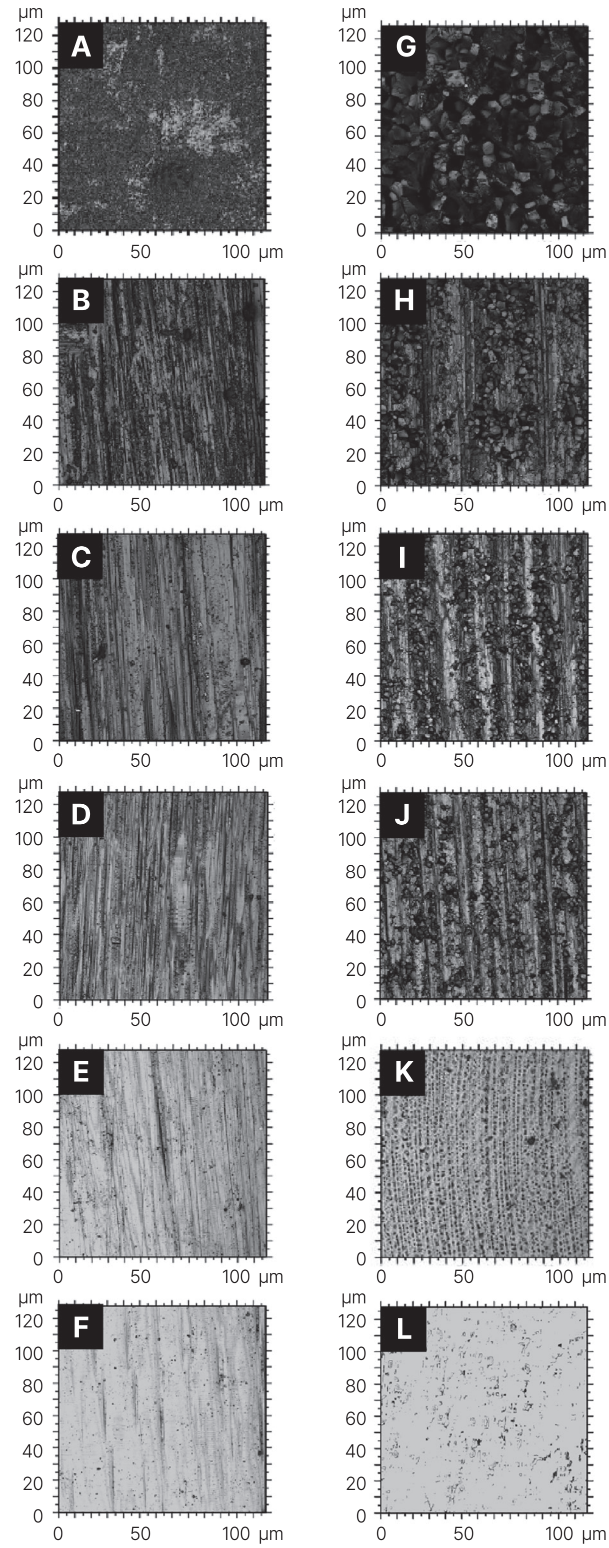

Fig. 4. Confocal laser microscopy images of specimens subjected to different finishing and polishing procedures. LS group (A-F), CS group (G-L), as delivered state (A, G), blue ground state (B, H), red ground state (C, I), yellow ground state (D, J), Jota step 1 polished state (E, K), Jota step 2 polished state (F, L).

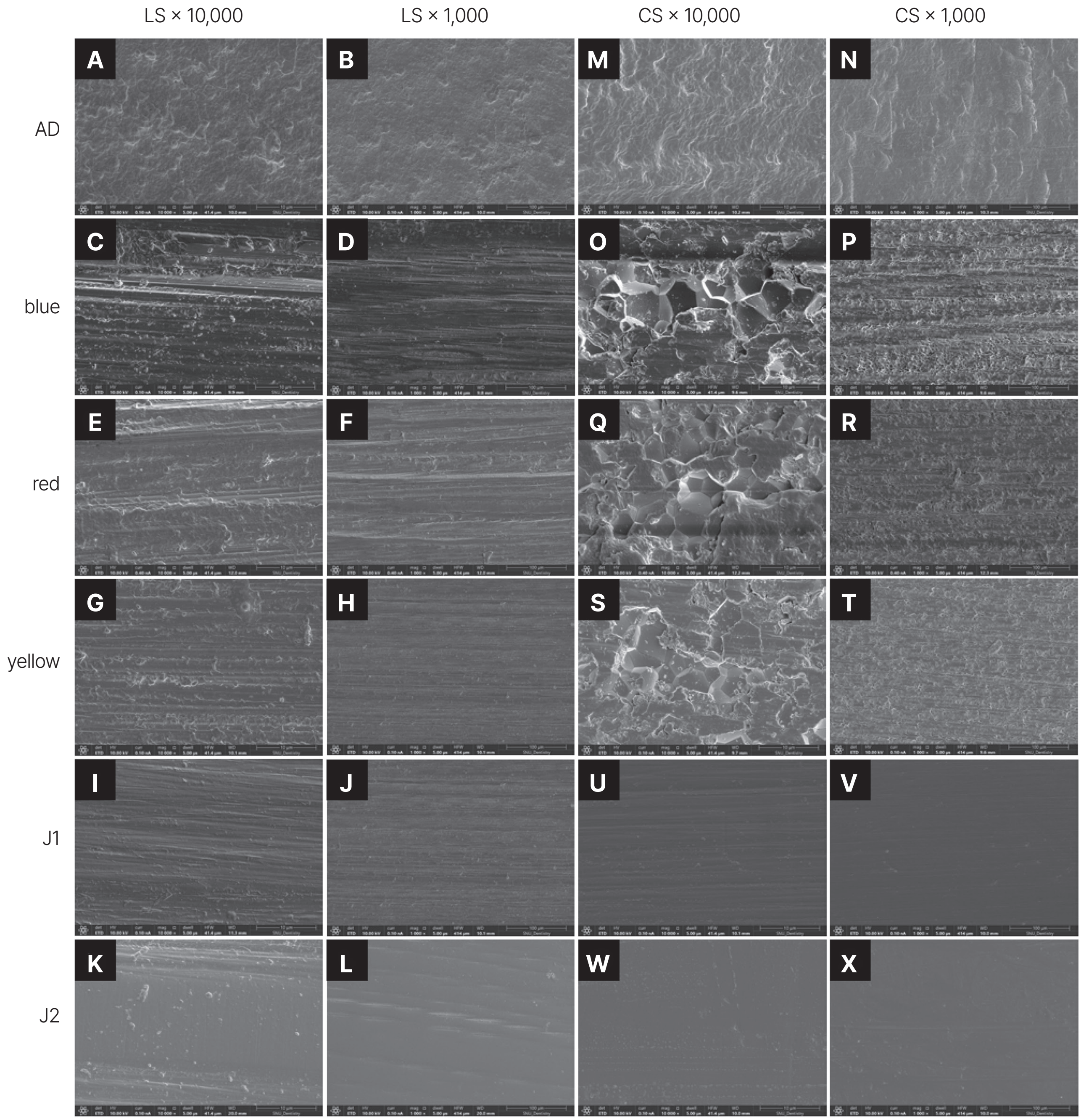

Fig. 5. Scanning electron micrographs (magnification ×1,000 and ×10,000) of specimens subjected to different finishing and polishing procedures. Groups and subsequent treatments are notated on top and left row of the figure, respectively. AD, as delivered state; blue, blue diamond bur ground state; red, red diamond bur ground state; yellow, yellow diamond bur ground state; J1, Jota kit step 1 polished state; J2, Jota kit step 2 polished state; LS, labside; CS, chairside.

could be delivered in their milled form. Still, this hypothesis does not seem to be the case since CS blocks were milled using powerful milling machines, with phase transformation and rough surfaces appearing inevitable in their 'as-milled' state. Consequently, delivering these restorations in 'as-milled' or 'high-speed ground' states is not recommended.

Compared with other porcelain-based restorations, polished 3Y-TZP have been shown to cause less wear of opposing enamels.[17-19] In this study, CS

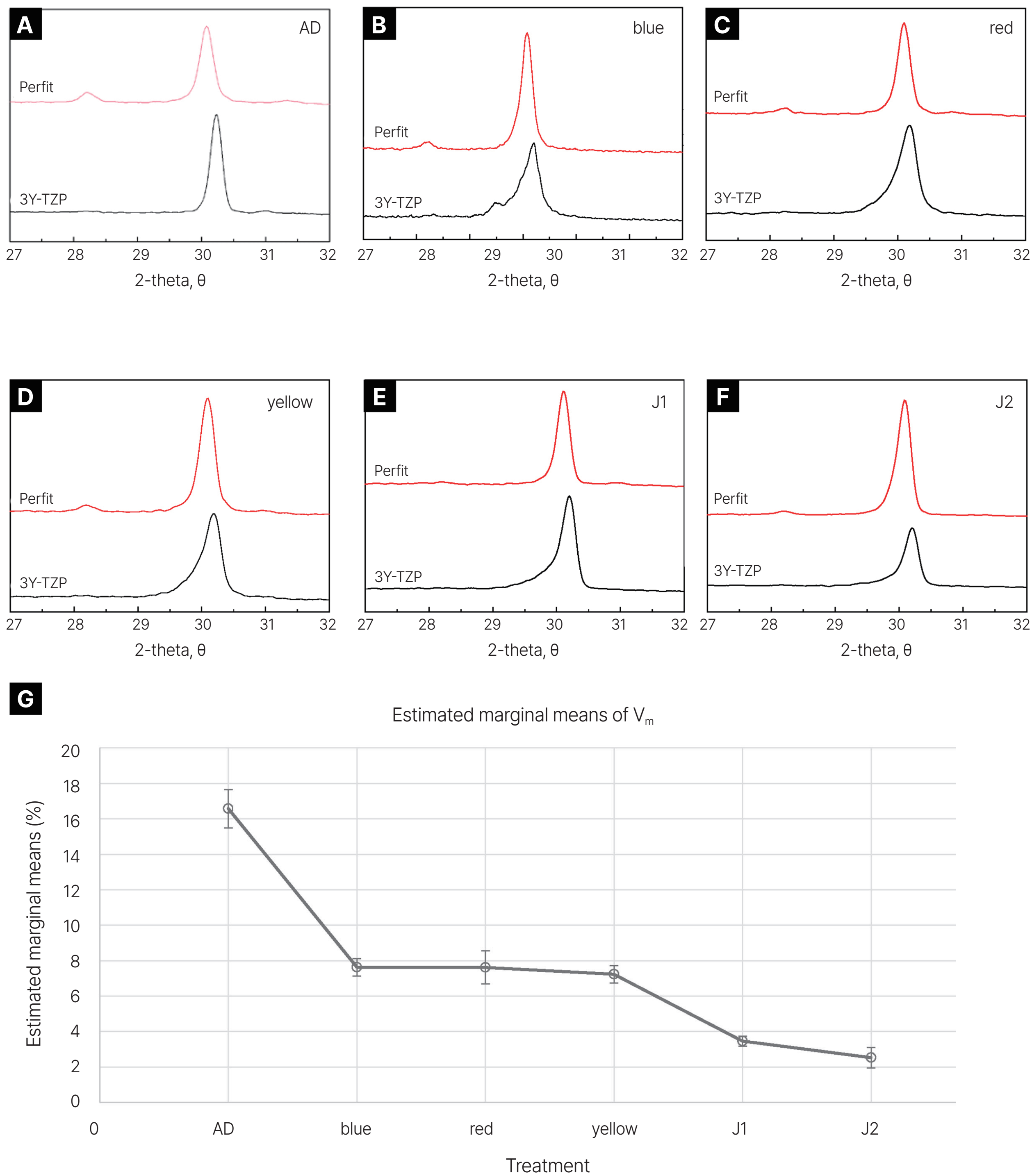

Fig. 6. XRD patterns and volume fraction values of m-ZrO_2 content, according to each surface treatment. (A-F) XRD patterns of each surface treatment, (G) estimated marginal means of volume fraction (%) values for each surface treatment. AD, as delivered state; blue, blue diamond bur ground state; red, red diamond bur ground state; yellow, yellow diamond bur ground state; J1, Jota kit step 1 polished state; J2, Jota kit step 2 polished state; LS, labside; CS, chairside.

group, when polished with conventional 2-step zirconia polishing kit, showed equivocal levels of surface roughness compared to conventional 3Y-TZP (LS). Therefore, since CS grinding and polishing is an inevitable step in the zirconia restoration delivery phase, it would be logical for clinicians to perform whole-round surface polishing of CS (Y, Nb)-TZP materials with the conventional 2-step zirconia polishing kit on delivery days.

The clinical implication of a phase transformation is a disputed topic, with some studies considering it a crack initiation site; and others considering it a protective stress layer that protects the prosthetic integrity.[22,35-37] Nevertheless, while the clear clinical effect of a phase transformation is yet to be concluded, it is a generally accepted idea that the regular thin, even layer of monoclinic zirconia is preferable compared to the thick, irregular transformed layer.[38] Therefore, the fine polishing technique adopted in our study would be a clinically suggestible procedure in handling the Perfit-FS restorations.

Currently, there is no strict research protocol for zirconia surface grinding and polishing studies. Some studies use a machinery-driven polishing apparatus on flat zirconia specimens,[36, 39-41] with defects that are different from those created during clinical practice being introduced by these types of machinery.[40,41] These observations propose that in clinical situations, many aspects could change the characteristics of the polished surface.[17,42-44] To this end, our study created a crown specimen to simulate clinical polishing procedures. Still, due to this study's design, it was impossible to directly compare numerical values, such as the Ra values of our study to previous studies. Furthermore, because of this study design, it was not possible to assess mechanical properties, such as flexural strength or surface hardness, as it requires standardized specimen preparations, such as disks or blocks. Our previous studies on mechanical data of CS (Y, Nb)-TZP were performed on as-milled specimens.[16] Thus, surface treatment may increase mechanical values because polishing procedures were proposed to increase mechanical characteristics, such as flexural strength.[21,45] However, this hypothesis remains speculative.

One major issue encountered regarding zirconia restoration is decementation and glaze chipping.[46] Presumably, reduced internal surface roughness and lack of micromechanical bonding may cause such detachments.[47] With that fact in mind, rougher surfaces of milled CS zirconia blocks, under the assumption that they could be finely polished, could have advantages in both cementation and glazing properties. In our pilot study, the M phase transformation was not visible in CS (Y, Nb)-TZP after regeneration firing (data not shown). Thus, annealing may be considered for this restoration. These should be addressed in future studies.

CONCLUSION

The CS (Y, Nb)-TZP zirconia showed significantly higher Ra values and volumetric M phase contents than the conventional 3Y-TZP zirconia in the AD and ground states. In addition, the CLSM and FE-SEM images confirmed grain exposure of the milled and ground surfaces. Therefore, we polished CS (Y, Nb)-TZP zirconia with a conventional zirconia polishing kit. As a result, the roughness of zirconia crowns was reduced to equivocal levels of the polished 3Y-TZP. Fine polishing also resulted in reduced levels of phase transformation. Hence, our results confirm the validity of grinding and polishing procedures for zirconia restoration, as suggested by previous studies. In clinical situations, adjustment with high speed diamond bur, followed by sequential polishing with conventional zirconia polishing kit, can be recommended for (Y, Nb)-TZP restorations as well.

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