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Original Article

The comparisons of layers and the effect of additional firings on flexural strength and translucency of 5Y-ZP

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Purpose: The purpose of this in vitro study was to investigate the flexural strength and translucency of three layers in 5Y-ZP and to assess the effect of additional firings on these properties. **Materials and Methods:** Sintered zirconia blocks were sectioned according to three layers : incisal, transition, and body. Disc-shaped specimens were fabricated from each layer. The diameter of specimens was 15.0 mm and each thickness of specimens for biaxial flexural strength test and translucency was 1.2 mm and 1.0 mm. The specimens were classified into subgroups according to the number of firing (0, 1, and 3 times; n = 10/subgroup) and the additional firings were performed under 900°C using a furnace. Biaxial flexural strength and translucency was measured using universal testing machine and uv-vis spectrophotometer. X-ray diffraction (XRD) analysis was used for measurement of the phase identification. One-way ANOVA, Tukey HSD test were performed ($\alpha = 0.05$). **Results:** There was no significant difference in flexural strength between the three layers ($P > 0.05$), while there was significant difference in translucency between different layers ($P < 0.05$). The flexural strength of incisal and transition layer was decreased by the single additional firing, and the three additional firings significantly decreased the flexural strength of three layers. The translucency of layer was decreased by additional firings except the body layer. The XRD patterns of all groups were similar. **Conclusion:** Three layers of 5Y-ZP were different only in translucency. Additional firings affected the flexural strength and translucency differently depending on the layers but crystalline phases were not changed. **(J Dent Rehabil Appl Sci 2021;37(3):111-22)**

Key words: monolithic zirconia; cubic phase; flexural strength; translucency; 5Y-ZP

Introduction

Metal-ceramic, glass-ceramic, and zirconia restorations have been used in dentistry for esthetic outcomes. Metal-ceramic materials have shown successful mechanical properties, however, they have esthetic limitations such as exposure to metal and gingival discoloration.[1] Glass-ceramic materials show high translucency and advantages in esthetics, but those are vulnerable to fracture due to weak mechanical properties.[2,3] Zirconia has good mechanical properties and biocompatibility,[4] however, because of its intrinsic opacity, it is usually used with porcelain veneers. The major complication of zirconia-framework restorations is the high failure rate associated with porcelain chipping and fracture.[5,6]

Monolithic zirconia has been used to prevent porcelain chipping and fracture.[7-9] Zirconia is a polymorphic material with three crystalline phases: monoclinic, tetragonal, and cubic. Pure zirconia is stable in monoclinic phase at room temperature and transforms into tetragonal phase at 1170°C. Further,

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at 2370°C, the cubic phase is stable. As zirconia cools down to room temperature, the transformation from tetragonal to monoclinic (t-m transformation) occurs with volume expansion inducing crack formation. The addition of stabilizer is essential to prevent the cracks. In dentistry, 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is most widely used. The metastable tetragonal phase at room temperature is facilitated by doping 3 mol% yttria as stabilizer. The 3Y-TZP exhibits a special mechanism known as transformation toughening. The stress induces t-m transformation and the volume expansion generates the compressive stress which resists crack propagation. Based on the mechanism, 3Y-TZP has an approximate flexural strength of 900 to 1200 MPa and a fracture toughness of 9 to 10 MPa/m^2. In the aspect of mechanical properties, monolithic zirconia is useful under defective interocclusal clearance or when less invasive preparation is needed.[10,11] However, it is usually restricted to posterior teeth because of low translucency.[12]

Recently, to overcome the esthetic limitation, the translucency of zirconia was improved by increasing the yttria content. Doping zirconia with 5 mol% yttria creates a partially stabilized zirconia containing approximately 50% cubic phase.[13] The cubic phase of zirconia has isotropic refractive index, which decreases the light scattering and the birefringence phenomenon,[11,14] while the tetragonal phase of zirconia has anisotropic refractive index. As a result, the 5 mol% yttria-stabilized zirconia polycrystal (5Y-ZP) shows higher translucency compare to the conventional 3Y-TZP.[14] Despite the improved translucency, the mechanical properties of 5Y-ZP are lower than those of 3Y-TZP due to lack of transformation toughening. Thus, 5Y-ZP is susceptible to damage because the stabilized cubic phase does not transform at room temperature.[13-15]

Many 5Y-ZP blocks, which are also known as translucent zirconia, are available commercially. According to the manufacturers' report, the flexural strength of 5Y-ZP blocks is approximately 600 to 750 MPa and the translucency of 5Y-ZP blocks is improved. Furthermore, some of the 5Y-ZP blocks contain a pre-shaded multi-layers to reproduce the shades of natural teeth.[16]

Many studies report the mechanical properties and the translucency of 3Y-TZP[3,4,17-20]; however, studies related to the mechanical properties and translucency of 5Y-ZP are lacking. If the mechanical properties and the translucency of layers are different, those of fixed prosthesis will depends on how the position of prosthesis in the 5Y-ZP block is determined by computer-aided design. The design of prosthesis should be different considering the clinical situations such as the natural shade of teeth, occlusion and interocclusal clearance. It is important to find out the difference of layers in 5Y-ZP but few studies have compared flexural strength and translucency between layers of 5Y-ZP.[21,22] Despite the shade of 5Y-ZP matches the classical shade guide, additional external staining is essential because the shade of natural teeth is variable. The manufacturers recommend firing under 900°C, but the effect of additional firing on 5Y-ZP is unclear. This study aimed to investigate the biaxial flexural strength and translucency of different layers in 5Y-ZP and to assess the effect of additional firing on the flexural strength and translucency. The null hypotheses are (1) that there is no difference in the flexural strength and translucency of the different layers in 5Y-ZP and (2) that additional firing does not affect the flexural strength and translucency.

Materials and Methods

Specimen preparation

5Y-ZP block (Lava$^{\text{TM}}$ Esthetic Fluorescent Full-Contour Zirconia, 3M ESPE, Seefeld, Germany) containing a pre-shaded multi-layer, was selected. Blocks of pre-sintered zirconia were sintered according to sintering protocols summarized in Table 1. After sintering, the blocks were sectioned according to three different layers: incisal, transition, and body layer. Disc-shaped specimens measuring 15.0 mm in diameter were cut from each layer using a surface grinding machine (HRG-150, AM Technology, Asan, Korea) and mirror-polished with diamond particles measuring up to 1.0 μm using a grinding machine

Table 1. Sintering protocol

	High temperature	Hold time	Rate of temperature increase	Rate of temperature decrease
Lava^{TM} Esthetic	1,500°C	120 minutes	20°C/min to 800°C 10°C/min to 1,500°C	-15°C/min to 800°C -20°C/min to 250°C

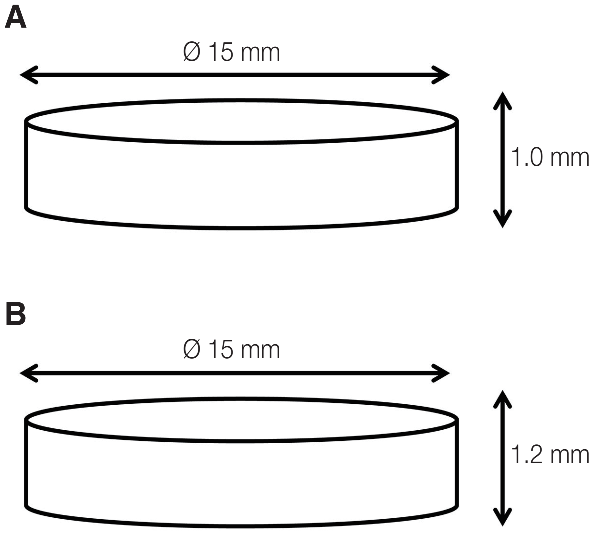

Fig. 1. The disc-shaped specimens. (A) Translucency test, (B) Biaxial flexural strength test.

(SPL-15 Grind X, OKAMOTO Co., Tokyo, Japan) to generate thicknesses of 1.0 mm in the translucency test and 1.2 mm in the biaxial flexural strength test (Fig. 1). The final dimensions of the specimens were measured with a digital caliper. The specimens were cleaned in distilled water for 10 min using ultrasonic cleaner (SD-120H, Mujigae Co, Seoul, Korea) and air-dried for 20 s. The specimens of each layer for biaxial flexural strength test (n = 30) and for translucency test (n = 30) were randomly classified into three groups according to the number of additional firings: 0, 1, and 3 times. The classification is shown in Table 2 and each group comprised 10 specimens.

The additional firings were performed using a furnace (Programat® EP 5000, Ivoclar Vivadent, Schaan, Lichtenstein) to reproduce external staining. The firing schedule was set based on the manufacturer' s recommendation for low-temperature firing under 900°C (Table 3).

Biaxial flexural strength test

Disc specimens (Ø = 15.0 mm, thickness = 1.2 mm) were fabricated according to ISO 6872:2015.[23] The piston-on-three-ball test was used according to ASTM Standard F394-78.[24] Three steel spheres (Ø = 3.2 mm) were positioned at 120° from each other on the perimeter of a circle with 10.0 mm diameter. The circular cylinder of hardened steel with a 1.6 mm diameter was used for loading (Fig. 2). The biaxial flexural strength was measured using a universal testing

Table 2. Classification of groups of tested specimens

Layers	Group code	Groups		
		Firing (n = 0)	Firing (n = 1)	Firing (n = 3)
Incisal layer	IL	IL0	IL1	IL3
Transition layer	TL	TL0	TL1	TL3
Body layer	BL	BL0	BL1	BL3

Table 3. Additional firing schedule

Dry-out time	Predrying temperature	Heat rate	High temperature	Cool time
5 min	600°C	50°C/min	850°C	4 min

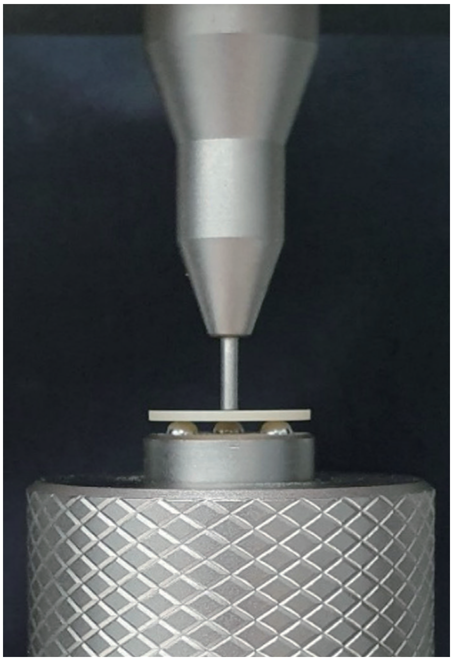

Fig. 2. The piston-on-three-ball test.

machine (QM100TS, QMESYS, Seoul, Korea) at a 1.0 mm/min crosshead speed. The specimens were subjected to stress until fracture occurred and the load value causing fracture (N) was recorded. The biaxial flexural strength was calculated using the following equation (1).

(1) $S = -0.2387P(X - Y) / b^2$

where: S; maximum center tensile stress (MPa), P; total load causing fracture (N), b; thickness of the specimen (mm). X and Y were determined using the following equation (2) and (3).

(2) $X = (1 + v) \ln \left(\frac{r_2}{r_3}\right)^2 + \left[\frac{1 - v}{2}\right] \left(\frac{r_2}{r_3}\right)^2$

(3) $Y = (1 + v) \left[1 + \ln \left(\frac{r_1}{r_3}\right)^2\right] + (1 - v) \left(\frac{r_1}{r_3}\right)^2$

where: v; Poisson's ratio, r_1; radius of the support circle (mm), r_2; radius of the loaded area (mm), r_3; radius of the specimen (mm). In the present study, the parameters v, r_1, and r_2 were set to 0.33, 5.0 mm, and 0.8 mm, respectively.

Translucency

Disc specimens (Ø = 15.0 mm, thickness = 1.0 mm) were fabricated to measure the transmittance of light. A uv-vis spectrophotometer (UV-2600, SHIMADZU, Tokyo, Japan) with an integrating sphere was used to evaluate the total transmittance of light in percentage (T_t) calculated using the following equation (4) and (5).

(4) $T_t = \frac{I}{I_0}$

(5) $T_t\% = T_t \times 100$

where: T_t; total transmittance of light, I; intensity of the light after its transmission through the specimen, I_0; initial light intensity.

Measurement conditions were set as follows: wavelength range of 380 to 780 nm, a data interval of 1 nm, and a deuterium light source. The translucency was compared by selecting the mean $T_t\%$ values at the wavelength of 555 nm. Since the eye is most sensitive to the wavelength of 555 nm, the International Commission on Illumination (CIE S 017) and the Japanese industrial standard (JIS Z 8113) defined the wavelength of 555 nm as the maximum luminous intensity.[25,26]

X-ray diffraction

X-ray diffraction(XRD) analysis was used to evaluate the changes in phases after additional firing. The two specimens of each group were randomly selected and Cu Kα (40 kV, 40 mA) XRD analysis was performed using a diffractometer (D8 ADVANCE; Billerica, Massachusetts, USA). Each specimen was analyzed from 20° to 90° 2θ with a step size of 0.02°.

Statistical analysis

The statistical analysis was conducted with IBM SPSS Statistics v24.0 (IBM Corp., Chicago, USA). The significance of the differences in flexural strength and translucency among the groups were analyzed by Kolmogorov-Smirnov Test, and one-way ANOVA. A post-hoc analysis was conducted via Tukey HSD test. The effect of additional firing on the flexural strength and translucency was analyzed using the same methods. The level of statistical significance was set at 5%.

Results

Body layer showed the highest value and incisal layer showed the lowest value of biaxial flexural strength, but there was no significant difference among three layers ($P > 0.05$). Additional firing had significant effect on biaxial flexural strength of each layers. IL0 showed significantly higher flexural strength than IL1 ($P = 0.033$) and IL3 ($P = 0.018$), but there was no significant difference between IL1 and IL3 ($P > 0.05$). Among the transition layer groups, TL0 showed significantly higher flexural strength than TL1 ($P = 0.022$) and TL3 ($P = 0.004$). However, no significant difference between TL1 and TL3 was observed ($P > 0.05$, Fig. 3). BL0 showed the highest biaxial flexural strength but significant difference was detected only between BL0 and BL3 ($P = 0.003$).

Unlike the flexural strength, the translucency of three layers showed significant difference.

The incisal layer was significantly more translucent than other layers ($P < 0.05$) and the body layer was significantly less translucent than other layers ($P < 0.05$, Fig. 4, Table 5).

IL3 group was less translucent than IL0 ($P < 0.001$) and IL1 ($P < 0.001$), but IL0 and IL1 showed no significant difference ($P > 0.05$). TL0 showed the highest value and TL3 showed the lowest value of transmittance among the transition layer groups ($P < 0.001$). In contrast to other layers, there was no significant difference of transmittance after additional firings in body layer ($P > 0.05$).

The total transmittance of each layer from 380 to

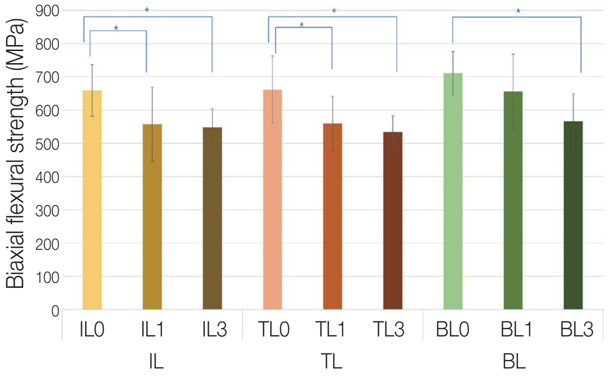

Fig. 3. Mean biaxial flexural strength.
* denotes significant difference at the level of 0.05.

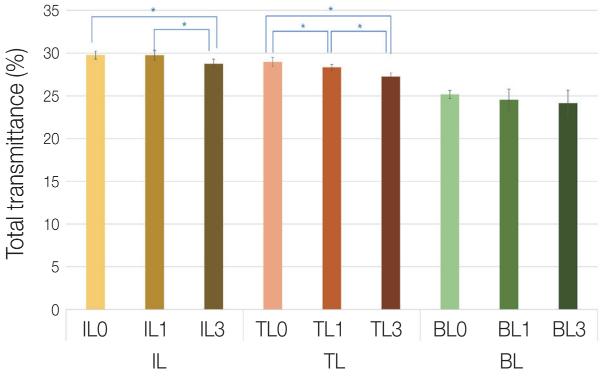

Fig. 4. Mean total transmittance of 555 nm wavelength.
* denotes significant difference at the level of 0.05.

Table 4. Mean values and standard deviations of biaxial flexural strength (MPa)

Group	Mean (SD)		
	Firing (n = 0)	Firing (n = 1)	Firing (n = 3)
IL	658.98 (77.65)	557.48 (111.60)	547.57 (55.86)
TL	661.62 (101.31)	559.21 (82.23)	534.17 (49.11)
BL	710.86 (65.10)	655.65 (112.56)	566.13 (82.93)

SD: standard deviation, IL: Incisal layer, TL: Transition layer, BL: Body layer.

Table 5. Mean values and standard deviations of total transmittance (%) measured at 555 nm wavelength

Group	Mean (SD)		
	Firing (n = 0)	Firing (n = 1)	Firing (n = 3)
IL	29.76 (0.47)	29.74 (0.58)	28.77 (0.53)
TL	28.97 (0.53)	28.34 (0.32)	27.26 (0.42)
BL	25.17 (0.47)	24.55 (1.24)	24.14 (1.53)

SD: standard deviation, IL: Incisal layer, TL: Transition layer, BL: Body layer.

780 nm wavelength was shown in Fig. 5. The total transmittance showed an upward tendency as the wavelength increased; however, all layers exhibited a similar steep decline from 480 to 490 nm, 500 to 525 nm, 540 to 543 nm, and 637 to 655 nm wavelength.

The peak of the x-ray diffraction was observed in all groups around 30°, 35°, 50°, 59°, 62°, 74°, 81°, and 84° 2θ showing similar patterns (Fig. 6). Definitive phase transformations were not observed after additional firings in all three layers.

Discussion

In this study, the differences in flexural strength and translucency between different layers of pre-shaded 5Y-ZP and the effect of additional firings on flexural strength and translucency of 5Y-ZP were tested. The flexural strength of different layers was similar, despite significant difference in translucency between different layers. Therefore, the first null hypothesis was partially rejected. The second null hypothesis that additional firing does not affect the flexural strength and translucency of 5Y-ZP was also rejected.

Biaxial flexural strength test does not show edge failure and the effect of crack direction is minimal, and therefore, is considered more accurate than uniaxial flexural strength.[27,28] Thus, the flexural strength was measured using biaxial test and approximately 650 to 750 MPa was measured in groups without additional firing. The result was similar to the flexural strength of another pre-shaded 5Y-ZP reported by Kwon et al.[14] Although the 5Y-ZP showed a lower flexural strength than that of 3Y-TZP due to the lack of transformation toughening, it yielded higher flexural strength than lithium disilicate.

The disc-shaped specimens were fabricated after the sintering of 5Y-ZP because the pre-sintered blocks were vulnerable, and the boundaries of three layers were difficult to distinguish completely before sintering due to ambiguous color differences. The unintentional stress may have occurred during fabrication of specimens but the result was in accordance with the flexural strength of other 5Y-ZP.

The layers of 3Y-TZP showed significantly different flexural strength,[29] whereas the layers of 5Y-ZP did not show significant differences despite the slight increase in flexural strength from the incisal layer to the body layer. It is difficult to explain the factors underlying the differences in results but the type and

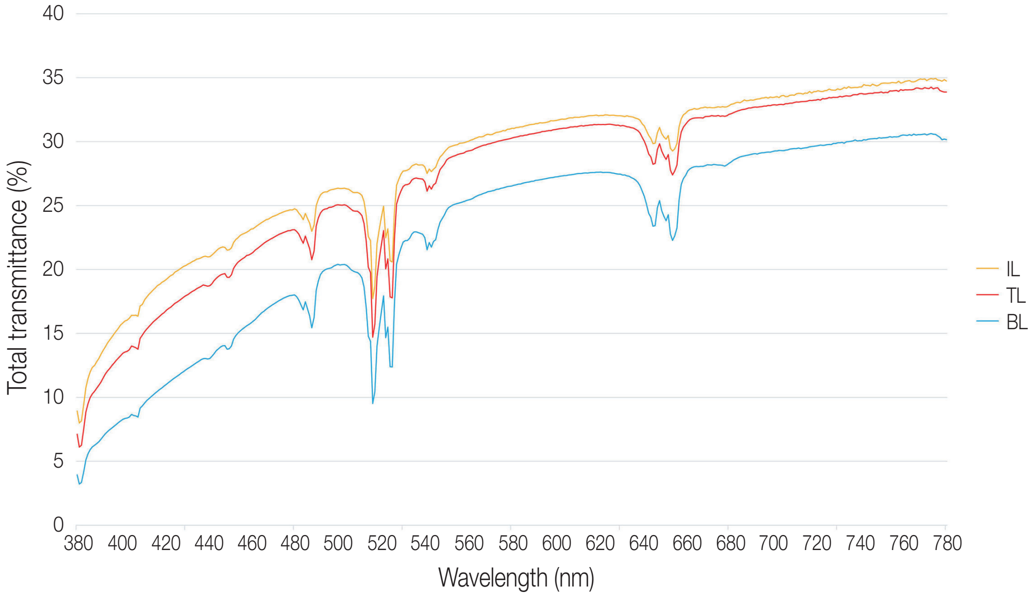

Fig. 5. Total transmittance spectrum for each layer.
IL: Incisal layer, TL: Transition layer, BL: Body layer.

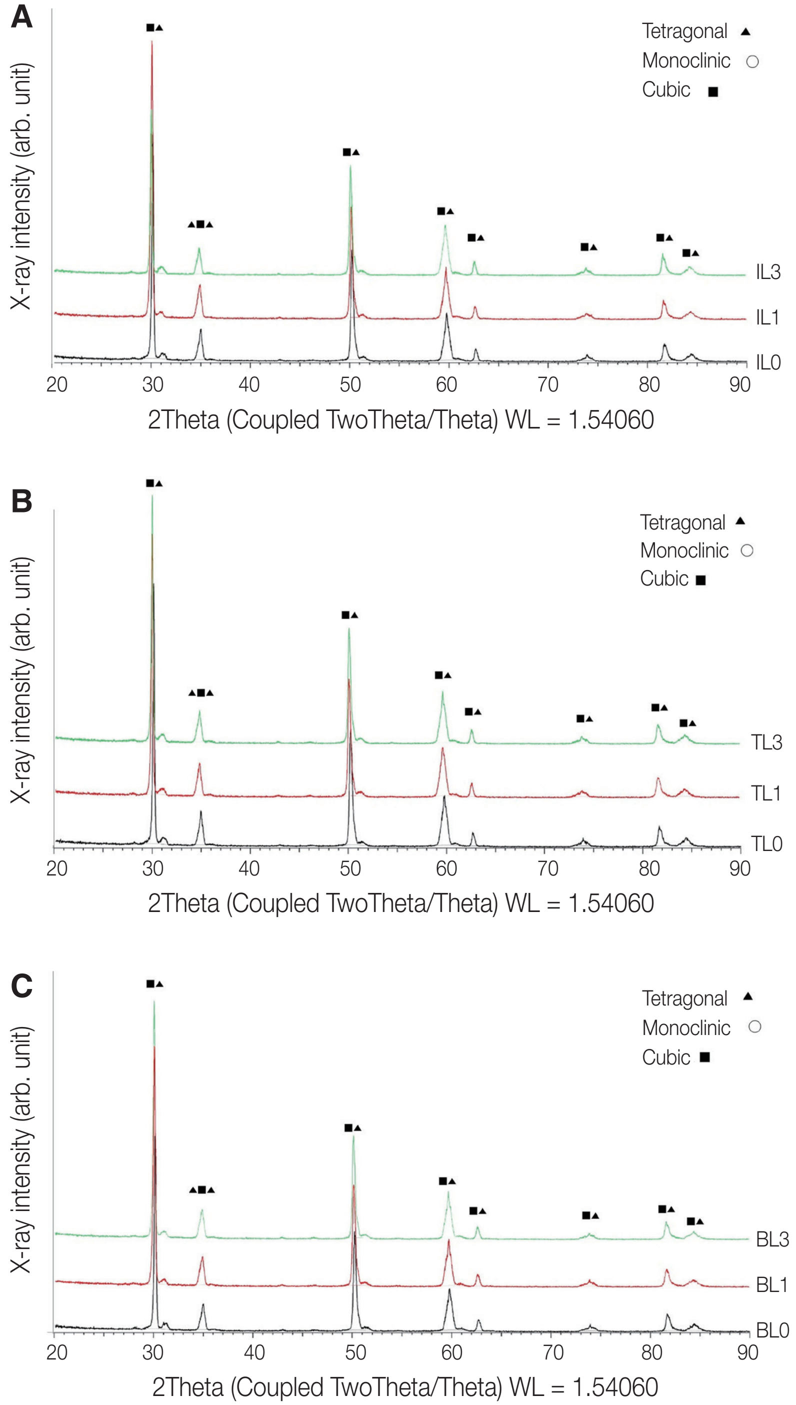

Fig. 6. Representative XRD patterns of 5Y-ZP. (A) Incisal layer groups, (B) Transition layer groups, (C) Body layer groups.

concentration of shading gradients may influence the strength of zirconia.[20] Moreover, since the cubic phase is increased and lacks transformation toughening, the difference between layers might be minimal. The failure rates of fractures due to different flexural strength between layers may be reduced in 5Y-ZP.

Five veneering firing cycles did not affect the flexural strength of 3Y-TZP and the number of firing cycles had negligible effects on the flexural strength of 3Y-TZP.[30,31] In contrast, the additional firing decreased the flexural strength of 5Y-ZP. Three additional firings significantly decreased the flexural strength of the three layers and the single additional firing decreased the flexural strength of incisal and transition layers. The factors underlying the differential effects of additional firing on 5Y-ZP compared with that of 3Y-TZP are unclear; however, the microstructure of zirconia might influence the response of zirconia to the heat. The firing may also lead to changes in the shape of porosities and impurities embedded in the specimens, facilitating crack propagation and decrease in the flexural strength.[32] The single additional firing decreased the flexural strength of 5Y-ZP and the three firings resulted in a detrimental effect, and therefore, not recommended for staining. If the portion of the incisal and transition layer is increased, even the single additional firing should be done with consideration.

There are various methods to measure the translucency of materials: contrast ratio (CR), translucency parameter (TP), direct transmittance (T_d%) and total transmittance (T_t%).[33-36] The CR is a measure of the ratio of reflectance from a material on a black background to the reflectance on a white background. The TP refers to the color difference of material between black and white backgrounds. Measurement of T_t% is appropriate for transparent or clear materials because of minimal scatter or diffusion of light. Instead, when measuring the translucency of translucent or hazy materials such as dental ceramics, it is more appropriate to measure the TP because it indicates not only the direct transmittance but also diffuse transmittance.[33,36,37] Also, the CR is not a direct method for measuring translucency and should not be used below 50% transmission.[38] Therefore, the T_t% was measured to evaluate the translucency.

The translucency of various 5Y-ZP was reported in current studies.[39,40] In this study, the translucency of 5Y-ZP was lower than the results of other studies. In contrast to other pre-shaded 5Y-ZP which uses 2 or 3 shading gradients, the 5Y-ZP used in this study consists of 4 shading gradients, which include the fluorescent shading element for representing the fluorescence of natural teeth. Increased number of shading gradients might decrease the translucency. Another possible explanation is that the result showed a lower translucency due to the average grain size. The manufacturer reports that the average grain size of Lava™ Esthetic Fluorescent Full-Contour Zirconia (3M ESPE) was 0.7 μm, while the average grain size of another 5Y-ZP was 1.7 μm.[40,41] The larger the grain size is, the more translucent the zirconia becomes due to decrease of grain boundaries. Since the 5Y-ZP in the present study had a smaller grain size, the light scatter might increase.

The translucency of different layers of 5Y-ZP showed no significant difference in the previous studies,[21,22] but the studies measured the TP to evaluate translucency. However, the translucency was significantly different between layers based on T_t%. When designing the restoration using CAD, the apicocoronal positioning of the restoration should be determined carefully because of varying translucency of layers and diverse shade of natural teeth. If the shade of abutment is similar to that of adjacent teeth, positioning the design of the restoration coronally in the 5Y-ZP block will produce better outcome by reproducing the shade of natural teeth. However, if the shade of abutment is different from that of adjacent teeth and masking is needed, the restoration should be positioned apically. The additional firing significantly decreased the translucency of 5Y-ZP except the body layer. Therefore, in the aspect of esthetic outcome, three additional firings of 5Y-ZP are not recommended and the single additional firing should be considered carefully.

The total transmittance of each layer was different but the spectral patterns were similar to each other. The transmittance increased as the wavelength increased, but there were steep declines at 480 to 490 nm, 500 to 525 nm, 540 to 543 nm and 637 to 655 nm wavelength, consistent with blue, green, red colors. The 3 colors are the complementary colors of cyan, magenta, yellow, which are the basic colors of subtractive mixture. Therefore, the pre-shaded 5Y-ZP absorbed additional light at the specific wavelengths and decreased the translucency.

The three layers showed similar patterns of x-ray diffraction and the results were in accordance with the study of Inokoshi et al.[41] In addition, the XRD patterns of groups fired additionally were not different from those before the additional firing. There was no change in the crystallographic phase of 5Y-ZP after firing but the decrease of flexural strength and translucency occurred. Even though the phase transformation was not detected, the microstructure might be changed and the shading gradients might influence the 5Y-ZP due to additional firing.

The number of additional firings was set according to the study measuring the color change of porcelain after repeated staining procedures. The study reported that the color change was perceptible after the staining initially with one type of stain and the color change was perceptible after third staining in four stains.[42] However, the limitation of this present study still remains that the gradual changes of flexural strength and translucency, which could appear in sequential increase in number of firings, could not be evaluated because the two additional firings were not carried out. A significant difference in translucency was found in incisal and transition layers between single additional firing and three such firings. The effect of sequential firings on 5Y-ZP should be further studied for clinical use.

The additional firing was conducted without staining to evaluate the effect of low-temperature firing on flexural strength and translucency. The actual external staining might be detrimental because the low-firing temperature generates a porous glaze that wets the zirconia[43] and the moisture results in grain faceting at the surface of zirconia.[30] External staining might induce opacity of the 5Y-ZP as it absorbs the stain due to the microstructure and grain boundary.[44] The light transmission may be decreased; however, further studies are needed to report the effect of external staining on translucency and flexural strength of 5Y-ZP, focusing on the change of microstructure and grain size of 5Y-ZP.

Conclusion

On the basis of this in vitro study, the following conclusions can be drawn:

- There was a difference only in translucency, not in flexural strength of three layers.
- Three additional firings significantly decreased the flexural strength of 5Y-ZP, and the flexural strength was decreased in incisal and transition layer by the single additional firing. The translucency of transition layer was decreased by the single additional firing and the translucency was decreased in incisal and transition layers by the three additional firings, while there was no significant difference following additional firing in the body layer.
- There was a significant difference in biaxial flexural strength and translucency after additional firing but no changes in crystalline phases were observed.

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Translucent zirconia의 layer 간 비교 및 추가적인 소성이 굽힘강도, 투과도에 미치는 영향

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목적: 5Y-ZP의 세가지 층 간 굽힘강도, 투과도를 비교하고 추가적인 소성이 미치는 영향을 연구하고자 하였다.
연구 재료 및 방법: 소결한 지르코니아 블록을 세가지 층에 따라 분리한 후 원형 디스크 시편을 제작하였다. 시편의 직경은 15.0 mm이며, 2축굽힘강도와 투과도를 측정하기 위한 시편의 두께는 각각 1.2 mm와 1.0 mm이다. 시편들을 추가적인 소성 횟수(0, 1, 3번)에 따라 세 군으로 분류한 후 추가적인 소성은 900°C 이하의 온도에서 퍼니스를 사용하여 소성 횟수에 따라 시행하였다. 만능재료시험기와 자외선-가시광선 분광광도계를 사용하여 2축굽힘강도와 투과도를 측정하였다. 상의 변화를 관찰하기 위해 X선 회절 분석을 시행하였다. 측정값은 One-way ANOVA, Tukey HSD test를 통하여 분석하였다($\alpha = 0.05$).
결과: 층 간 2축굽힘강도는 유의한 차이가 없었지만($P > 0.05$) 투과도는 유의한 차이가 있었다($P < 0.05$). 절단 및 전이층은 1번의 추가적인 소성 후 굽힘강도가 유의하게 감소하였으며, 3번의 추가적인 소성 후에는 모든 층의 굽힘강도가 소성 전과 비교하여 유의하게 감소하였다. 몸체 층을 제외한 나머지 층들은 추가적인 소성 후에 투과도가 유의하게 감소하였다. 모든 그룹의 X선 회절 분석 결과는 유사하였다.
결론: 5Y-ZP의 세 층은 투과도의 차이만 존재하였다. 추가적인 소성은 각 층의 굽힘강도와 투과도에 다른 영향을 미쳤으나 상전이는 발견되지 않았다.

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주요어: 단일 구조 지르코니아; 입방정계; 굽힘강도; 투과도; 5Y-ZP

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