Optical properties and flexural strength of translucent zirconia layered with high-translucent zirconia

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This study investigated the optical properties and flexural strength of translucent TZP layered with high-translucent PSZ using resin cement of various shades. Zirconia specimens (translucent TZP; Zpex and high-translucent PSZ; ZpexSmile) were 13 mm in diameter, layered at thickness ratios of 0.3/0.7, 0.5/0.5, and 0.7/0.3 mm (ZpexSmile/Zpex), and then luted using resin cement of 3 shades. Monolithic specimens of both were used as controls. CIE L*a*b* color coordinates and translucency parameter (TP) were evaluated as optical properties. Biaxial flexural strength was also determined as a mechanical evaluation. The a* and b* values of layered specimens varied depending on the shade of cement. TP values were not affected by shade of cement and thickness ratio. The biaxial flexural strength was intermediate value between both monolithic specimens. The layering method of zirconia with various translucencies using resin cement of different shades can improve color expression while maintaining clinically sufficient flexural strength.

Keywords: Translucent zirconia, Layering method, Resin cement, Optical property, Flexural strength

INTRODUCTION

All-ceramic restorations have been become more common over traditional porcelain fused cast alloy (PFC) restoration due to growing interest in metal-free treatment due to higher esthetic demands. The clinical application of tetragonal zirconia polycrystal (TZP), a ceramic material with exceptional strength, is becoming more prevalent along with advances in computer aided design/computer aided manufacturing (CAD/CAM) technology1).

Conventional TZP has remarkable mechanical properties; however, its stark white color and lack of translucency makes them difficult to replicate the look of natural teeth1-4). As a solution to bear resemblance to the color and translucency of natural teeth, feldspathic porcelain was veneered on TZP core5). However, higher frequencies of chipping have been reported for all-ceramic restorations with TZP core compared to PFC restorations6-8). For this reason, monolithic TZP restorations, fabricated solely from TZP, were introduced; however, the low translucency of TZP limits its application in areas of high esthetic demand. Since its introduction, the composition, manufacturing process, and sintering methods of TZP have been modified in attempt to increase the translucency6-11).

In recent years, “translucent TZP” and “high-translucent partially stabilized zirconia (PSZ)” have become commercially available and its development has increased the scope of application for TZP restorations12). In addition to having higher strength and lower occurrence of chipping compared to porcelain fused TZP restorations, polished surface of TZP restorations causes less abrasion on antagonist teeth13,14).

The color of TZP restorations have been developed through changes in powder composition, coloring agents, pre-sinter staining, and post-sinter staining and glazing. Further esthetic improvements were made with multi-colored zirconia blocks, where TZP is horizontally added in layers from the cervical to incisal region to form a color gradation15). However horizontal layering method is still inadequate to reproduce the optical properties of actual enamel and dentin16).

In order to resemble color and translucency of natural tooth, materials should mimic anatomical structures of tooth such dentin and enamel. The translucency of dental ceramics is influenced by material thickness, number of firings, cement color, and lighting17-20). Malkondu et al. reported that restoration color can be adjusted with cement shade when translucent zirconia was used20,21). The use of resin cements to veneer leucite-reinforced glass ceramic on zirconia generated higher bond strength than the traditional fabrication method where glass ceramics were fired on zirconia22). Nevertheless, the effects of layering zirconia of various translucencies with cements on color, translucency, and mechanical properties have not been evaluated.

The aim of this study is to investigate the optical properties (color coordinates and translucency) and flexural strength of translucent TZP layered with high-translucent PSZ using resin cement of various shades. In this paper, we refer to translucent TZP layered with high-translucent PSZ as layered TZP.
MATERIALS AND METHODS

Specimen preparation

Translucent TZP (Zpex, Tosoh, Tokyo, Japan) and high-translucent PSZ (ZpexSmile, Tosoh) with translucent white color in diameter of 13 mm were used. Resin cement (Panavia V5, Kuraray Noritake Dental, Tokyo, Japan) in 3 shades (clear, universal, brown), and a functional monomer (10-methacryloyloxydecyl dihydrogen phosphate: MDP) containing primer (Ceramic primer plus, Kuraray Noritake Dental) were also used (Table 1).

Preparation procedures of layered and monolithic specimens are shown in Fig.1. Both Zpex and ZpexSmile discs were prepared from fully sintered blocks by cutting with a diamond wheel using a finecutter (HS-100, Heiwa Technica, Kanagawa, Japan). Specimen surface was roughly polished with a polishing machine (AutoMet 250 & EcoMet 250, Buehler Japan, Tokyo, Japan) using a diamond disc (Apex Diamond Grinding Disc-45 µm, Buehler, Lake Bluff, IL, USA).

Layered specimens were fabricated by adhered high-translucent ZpexSmile disc onto translucent Zpex disc in various thickness ratios. Before luting, the surface of ZpexSmile specimens was mirror polished with polishing cloth (TexMet, Buehler) and 3 µm diamond slurry (MetaDi 3 µm, Buehler). The adhesive surface of specimens and bottom surface of Zpex were sandblasted (Jet Blast, J. Morita, Tokyo, Japan) with 50 µm alumina (Al2O3) for 5 s under 0.3 MPa with the nozzle placed 10 mm from the center of the specimen at 90° incidence angle. After mirror polishing and sandblast treatment, surface roughness (Sa) was measured using 3D laser scanning microscope (3D-LSM, LEXT OLS4000, Olympus, Tokyo, Japan) with a measurement region of 645×644 µm². Sa values of mirror polished and sandblasted specimens were 0.05±0.01 and 0.35±0.03 µm, respectively. Final thickness of both Zpex and ZpexSmile specimens was adjusted to 0.30±0.02, 0.50±0.02, or 0.70±0.02 mm. After polishing and sandblast treatment, specimens were cleaned ultrasonically in acetone for 5 min. Adhesive surface of ZpexSmile and Zpex specimens was treated with MDP containing monomer, following manufacturer’s instructions, and luted at thickness ratios of 0.3/0.7, 0.5/0.5, and 0.7/0.3 mm (ZpexSmile/Zpex) by chemical curing under 50 N force for 10 min. The cement thickness was adjusted to 40 µm by using thickness gauge (THICKNESS GAUGE 172MA, Niigataseiki, Niigata, Japan).

As controls, monolithic specimens of Zpex and ZpexSmile 1.0 mm in thickness were prepared. Top surface was mirror polished and bottom surface was sandblasted. Specimens were stored in air and evaluated 24 h after luting.

Color analysis

Color analysis was performed using a colorimeter.

Table 1: Materials used in this study

| Materials               | Product name (Code) | Shade                  | Manufacturer                  | Lot no.       |
|-------------------------|---------------------|------------------------|-------------------------------|---------------|
| Translucent TZP         | Zpex® (Zpex)        | Translucent white      | Tosoh                         | Non           |
| High-translucent PSZ    | Zpex®Smile (ZpexSmile) | Translucent white     | Tosoh                         | Non           |
| Composite resin cement  | Panavia® V5         | Clear                  | Kuraray Noritake Dental       | 4D0007, 5W0007 |
|                         |                     | Universal              |                               | 4D0018, 6Q0035 |
|                         |                     | Brown                  |                               | 4D0004, 3F0004 |
| Ceramic primer          | Clearfil® ceramic primer plus | Non                 | Kuraray Noritake Dental       | 3P0015        |
The reflectance spectra at 8° against white and black backgrounds were determined to calculate the CIE $L^*a^*b^*$ color coordinates at CIE illuminant D65. $L^*a^*b^*$ values for white background were 98.0, 0.3, 1.7; $L^*a^*b^*$ values for black backing were 5.8, −0.5, −0.4.

Translucency was evaluated using the translucency parameter (TP) which is derived by calculating the difference between reflectance spectra against black (B) and white (W) background, because it was reported that TP corresponds directly to human visual perception of translucency so long as material thickness is equal\textsuperscript{23). TP was calculated using the following equation:

$$TP = [(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2}$$

Five specimens were used for each condition.

**Flexural strength**

For flexural strength test, specimens were placed so that the mirror-polished ZpexSmile was on the compressive (upper) side and sandblasted Zpex was on the tensile (lower) side of the jig (Fig. 2).

Biaxial flexural strength was determined using a universal testing machine (AUTOGRAPH AG-I 20 kN, SHIMADZU, Kyoto, Japan) with crosshead speed of 0.5 mm/min. Specimen indenter with a diameter of 1.5 mm was used.

Biaxial flexural strength was calculated in accordance with ISO 6872\textsuperscript{24) for both layered and monolithic specimens as monolayer specimens with following conditions according to manufacturer’s information: Young’s modulus 210 GPa and Poisson’s ratio 0.25 for Zpex and ZpexSmile\textsuperscript{25-27). Five specimens were used for each condition.

**SEM observation of fracture surface**

Fracture surface was ultrasonically cleaned with distilled water for 10 min, air dried, and carbon coated (Carbon Coater VC-100S, VACUUM DEVICE, Ibaraki, Japan) in preparation for observation under the scanning electron microscope (SEM SU6600, Hitachi, Tokyo, Japan).

**RESULTS**

**Optical properties**

Figure 3 displays the image of specimens observed under transmitted light. Background mark is visible in all specimens. In addition, differences in background was distinguishable between Zpex and ZpexSmile in monolithic specimens, even though it was hard to distinguish in layered specimens under the visible observation.

$L^*$ value showed significant differences in the 2 factors (cement shade and thickness ratio) and interaction between the 2 factors on two-way ANOVA under white and black backgrounds (Table 2). Monolithic Zpex specimens displayed the highest $L^*$ value on white background. No significant difference was found between all layered specimens and monolithic ZpexSmile specimens (Fig. 4a). On black background, monolithic Zpex specimens displayed the highest $L^*$ value and monolithic ZpexSmile specimens displayed the lowest. Layered specimens had $L^*$ values between the monolithic specimens (Fig. 4b).

The $a^*$ and $b^*$ values showed significant differences in the 2 factors (cement shade and thickness ratio) and
interaction between the 2 factors on two-way ANOVA under white backgrounds. Under black backgrounds, \( a^* \) and \( b^* \) values showed significant differences in the cement shade and interaction (Tables 3 and 4). The \( a^* \) and \( b^* \) values of layered specimens on white background were greater than monolithic specimens. For layered specimens, values were greater when brown cement was used and when thickness ratio of ZpexSmile was high (0.7/0.3, Figs. 5a and b). On black background, \( a^* \) and \( b^* \) had all negative values. Among them, monolithic ZpexSmile specimens displayed the lowest values (Figs. 6a and b). Both \( a^* \) and \( b^* \) values were greater against white background compared to black background; both showed similar tendency but \( b^* \) value displayed greater difference than \( a^* \).

TP values showed significant differences in thickness ratio on two-way ANOVA (Table 5).

Monolithic ZpexSmile specimens displayed the highest TP value at 21.3±0.6. No significant difference in TP value was found between layered specimens and monolithic Zpex specimen (15.0±0.6, Fig. 7).

**Biaxial flexural strength**

On two-way ANOVA, biaxial flexural strength showed significant differences in thickness ratio (Table 6). Monolithic Zpex specimens had the highest biaxial flexural strength at 1,038±115 MPa. Monolithic ZpexSmile had the lowest at 564±35 MPa. Flexural strength of layered specimens showed those values between two monolithic specimens, Zpex and ZpexSmile. On layered specimens, flexural strength did not significantly decrease even thickness ratio of ZpexSmile to Zpex was high (0.7/0.3, Fig. 8).

### Table 2 Results of 2-way ANOVA for \( L^* \) values

| White background | Sum of squares | df | Mean square | F     | \( p \) |
|------------------|---------------|----|-------------|-------|--------|
| Cement shade     | 8.91          | 2  | 4.46        | 5.69  | <0.01**|
| Thickness ratio  | 8.16          | 2  | 4.08        | 5.21  | <0.01**|
| Interaction      | 13.98         | 4  | 3.50        | 4.46  | <0.01**|

| Black background | Sum of squares | df | Mean square | F     | \( p \) |
|------------------|---------------|----|-------------|-------|--------|
| Cement shade     | 8.26          | 2  | 4.13        | 4.59  | 0.02*  |
| Thickness ratio  | 17.35         | 2  | 8.67        | 9.63  | <0.01**|
| Interaction      | 37.60         | 4  | 9.40        | 10.44 | <0.01**|

*: \( p<0.05 \), **: \( p<0.01 \)

Fig. 4 The \( L^* \) values on white background (a) and black background (b). Same lower-case letters indicate no statistical differences among the specimens (\( p<0.05 \)).
Table 3  Results of 2-way ANOVA for $a^*$ values

| White background | Sum of squares | df | Mean square | F   | p      |
|------------------|---------------|----|-------------|-----|--------|
| Cement shade     | 1.29          | 2  | 0.65        | 11.06 | <0.01** |
| Thickness ratio  | 0.52          | 2  | 0.26        | 4.48 | 0.02*  |
| Interaction      | 0.85          | 4  | 0.21        | 3.62 | <0.01** |
| Black background | Sum of squares | df | Mean square | F   | p      |
| Cement shade     | 0.17          | 2  | 0.08        | 5.29 | <0.01** |
| Thickness ratio  | 0.03          | 2  | 0.02        | 1.07 | 0.36   |
| Interaction      | 0.41          | 4  | 0.10        | 6.45 | <0.01** |

*: p<0.05, **: p<0.01

Table 4  Results of 2-way ANOVA for $b^*$ values

| white background | Sum of squares | df | Mean square | F   | p      |
|------------------|---------------|----|-------------|-----|--------|
| Cement shade     | 34.20         | 2  | 17.10       | 31.49 | <0.01** |
| Thickness ratio  | 4.75          | 2  | 2.38        | 4.37 | 0.02*  |
| Interaction      | 16.49         | 4  | 4.12        | 7.59 | <0.01** |
| black background | Sum of squares | df | Mean square | F   | p      |
| Cement shade     | 10.82         | 2  | 5.41        | 44.29 | <0.01** |
| Thickness ratio  | 0.53          | 2  | 0.27        | 2.18 | 0.13   |
| Interaction      | 5.72          | 4  | 1.43        | 11.71 | <0.01** |

*: p<0.05, **: p<0.01

![Graphs](image.png)

Fig. 5  The $a^*(a)$ and $b^*(b)$ values on white background. 
Same lower-case letters indicate no statistical differences among the specimens ($p<0.05$).

**SEM observation of fracture surface**

Figure 9 shows the SEM images of fracture surface after biaxial flexural test. Fracture of monolithic specimen seemed to start from the tensile side and travel towards the compressive side. For layered specimens, some fracture modes were similar to monolithic specimen. A
Fig. 6 The $a^*$ (a) and $b^*$ (b) values on black background. Same lower-case letters indicate no statistical differences among the specimens ($p<0.05$).

Table 5 Results of ANOVA for TP values

|                         | Sum of squares | df | Mean square | F   | p    |
|-------------------------|----------------|----|-------------|-----|------|
| Cement shade            | 1.14           | 2  | 0.57        | 0.67| 0.52 |
| Thickness ratio         | 7.15           | 2  | 3.58        | 4.20| 0.02*|
| Interaction             | 4.02           | 4  | 1.00        | 1.18| 0.34 |

*: $p<0.05$

Fig. 7 TP values. Same lower-case letters indicate no statistical differences among the specimens ($p<0.05$).

Fig. 8 Biaxial flexural strength. Same lower-case letters indicate no statistical differences among the specimens ($p<0.05$).
Table 6 Results of 2-way ANOVA for biaxial flexural strength

|                         | Sum of squares | df | Mean square | F   | p    |
|-------------------------|----------------|----|-------------|-----|------|
| Cement shade            | 16,914.64      | 2  | 8,457.32    | 2.14| 0.13 |
| Thickness ratio         | 28,701.24      | 2  | 14,350.62   | 3.63| 0.04*|
| Interaction             | 30,669.97      | 4  | 7,667.49    | 1.94| 0.13 |

*: p<0.05

DISCUSSION

In the present study, a thickness of 1.0 mm was selected for specimens. Heffernan et al. reported that increase in thickness of all-ceramic restorations decreases translucency, therefore restoration thickness must be carefully considered28). Sun et al. reported that for TZP, occlusal thickness of 1.0 mm can withstand as much load as PFC crowns29). Matsuzaki et al. reported that translucent TZP with 1.0 mm thickness showed a fracture load of 600 N, which is the clinical maximum occlusal load30). For these reasons, a thickness of 1.0 mm was selected for specimens used in this study.

Surface treatments were performed on specimens to assume clinical application. The surface of ZpexSmile was mirror polished as outer surface of restoration. The bottom surface of ZpexSmile and upper surface of Zpex were sandblasted and then applied primer. The bottom surface of Zpex was sandblasted to consider adhesion with abutment teeth. ZpexSmile surfaces were mirror polished because it has been reported that final polishing exhibits higher translucency compared to glazing treatment (firing with glaze and stain solution) in which an interface is formed31). For cement adhesion, sandblasting and application of functional monomers on TZP surface are recommended because improvements in mechanical interlocking is seen in the former and increased chemical luting is seen in the latter1,32,33).

representative image of the cement boundary is shown on Fig. 10. No signs of delamination at the cement interface was observed.
In this study, $L^*$ value was similar against white background for all specimens even though Zpex had the highest value. One possible explanation is considered that the color of Zpex and ZpexSmile used was translucent white, leading that $L^*$ value of the specimens was similar to the $L^*$ value of the white background. Differences in $L^*$ values were not seen for layered samples because of the same reason: the color of Zpex and ZpexSmile is white, thus a large difference in $L^*$ value was unlikely to occur in this condition. On the other hand, large deference in $L^*$ value was recognized between monolithic Zpex and ZpexSmile against black background, indicating the large difference in translucency between Zpex and ZpexSmile. Light scattering at the adhesive interface and blast surface should also be taken into consideration.

Our study showed $a^*$ and $b^*$ values of layered specimens displayed higher values than in both monolithic specimens; values were greater with increasing thickness ratio especially for cement in brown shade. These results indicated that color (hue and chroma) depends on the cement shade and as the ZpexSmile thickness ratio increases, the final color is more affected by the cement shade. In order to express color appropriately, the thickness of high-translucent ZpexSmile should be increased and cement shade should be selected carefully. In addition, change in $a^*$ and $b^*$ values with different shade was 1.9 and 7.7, respectively, in the present study. Asano reported that change in $L^*$ value was 1.9 and 7.7, respectively, be selected carefully. In addition, change in $ZpexSmile$ should be increased and cement shade should be selected carefully. In addition, change in $a^*$ and $b^*$ values with different shade was 1.9 and 7.7, respectively, in the present study. Asano reported that change in $a^*$ and $b^*$ values was 4.0 and 10.0 between A1 and A4 under a system of VITA classical shade, namely, 1.0 and 2.5 per one shade on average, respectively. Accordingly, the layering method of TZP with various translucencies using resin cement of different shades can improve color expression, though change in $a^*$ value is slightly small in this study. TP values for monolithic Zpex and ZpexSmile were 15.0±0.6 and 21.3±0.6, respectively. Layered specimens had similar TP values to monolithic Zpex. Since TP values of human dentin and enamel are reported to be 16.4 and 18.7, respectively, the translucency of monolithic Zpex and layered ZpexSmile/Zpex is similar to dentin, and translucency of monolithic ZpexSmile is similar to enamel. In this study, TP value of layered specimens demonstrated similar values to monolithic Zpex specimens. Translucency depends not only on material thickness but also on the scattering and absorption coefficient of the material. From this we can say that layered specimens have a multilayer and TP values of layered and monolithic Zpex specimens indicated no significant difference because of changes in light reflection and refraction mostly likely caused by surface roughening (sandblasting).

Biaxial flexural strength of Zpex and ZpexSmile was 1,038±115 and 564±35 MPa, respectively. These values are slightly lower than reported values, however it occurred most likely because the tensile side was sandblasted. Translucent TZP Zpex has lower content of Al$_2$O$_3$ (0.05 mass%) compared to conventional TZP to increase light transmittance. High-translucent ZpexSmile has higher content of yttria (Y$_2$O$_3$) and lower content of Al$_2$O$_3$ (0.05 mass%). In addition, cubic crystals coexist in the ZpexSmile, which dramatically improves translucency. Cubic crystals are optically isotropic and light scattering is small which increases translucency of sintered blocks, however strength decreases when cubic crystals are increased. Nevertheless, this material is resistant to low temperature degradation (LTD) because it contains high amounts of uniformly distributed Y$_2$O$_3$. During the flexural test, stress concentrates on the tensile side; therefore, strength was predicted to change due to the surface treatments performed on the surface of the tensile side. In this study, the surface of ZpexSmile (compressive side) was mirror polished and surface of Zpex (tensile side) was sandblasted but no significant differences in flexural strength was indicated on layered specimens. Biaxial flexural strength of layered specimens showed an intermediate value between Zpex and ZpexSmile. Despite the concern of reduction in strength which comes with decreasing the thickness ratio (ZpexSmile/Zpex=0.7/0.3), no decrease in flexural strength was observed. This is most likely because Zpex was placed on the tensile side, and stress concentration mostly occurred in the Zpex region with greater strength; therefore, overall strength was not influenced by ZpexSmile.

In the biaxial flexural test, strength is obtained as the tensile stress generated at the bottom center of the specimen when load is applied. When testing specimens layered with different elastic modulus, strength must be calculated using the elastic modulus of both materials. However, since the elastic modulus of both materials were 210 GPa, biaxial flexural strength of specimens were calculated as a single unified material. In addition, strength was calculated neglecting the existence of the cement layer assuming that the thin cement layer (4% of entire specimen) was firmly adhered. It was reported that porcelain veneered translucent TZP showed flexural strength of 500 and 150 MPa when thickness ratio of porcelain/Zpex was 0.5/1.0 mm and 1.0/0.5mm, respectively; strength dramatically decreased as porcelain thickness increased. Lithium disilicate glass-ceramic (IPS e.max) has been reported to have a flexural strength of 360–400 MPa. Layered specimens in this study had flexural strengths of 707±70 to 833±90 which is higher than the values derived in previous studies, therefore clinical application of restoration with thick ZpexSmile layer can be considered.

SEM images demonstrated differences in fracture mode of monolithic and layered specimens, however, delamination at the cement-zirconia interface was not observed regardless of thickness ratio. Layered specimens were unified through the use of resin cement even under the condition where ZpexSmile, with lower strength, was thick; and this observation is consistent with our results on flexural strength. Other studies have reported that when porcelain is veneered on TZP, fracture begins in the porcelain layer where it is weakest, then travels towards the interface where delamination occurs and induces fracture in the TZP core. On the
other hand, delamination and subsequent fracture was not observed in this study. One possible explanation of the differences is that the elastic modulus of both materials used for layering was 210 GPa; whereas, for the previous study, the elastic modulus of veneering porcelain was only 70 GPa and stress concentration in the weaker porcelain lead to fracture.

The results in the present study indicated that color expression of TZP crowns may be improved by luting high-translucent PSZ on translucent TZP using cements of various shades. In addition, translucent and high-translucent PSZ were bonded with resin cements, leading that porcelain firing was unnecessary which prevented deformation caused by thermal hysteresis. Furthermore, layered TZP, which was 1.0 mm in thickness, indicated clinically acceptable flexural strength; therefore, in comparison to PFC restorations, decrease in material thickness and tooth reduction is possible. However, the possibility of cement deterioration over time and LTD of TZP must be considered by performing the thermocycling test, and it is necessary to conduct a fatigue test in a simulated oral environment to examine whether strength can be maintained in the long term.

CONCLUSIONS

The following can be concluded from this study which investigated the optical properties (color coordinates and translucency) and flexural strength of translucent TZP layered with high-translucent PSZ using resin cement of various shades.

For optical properties, $a^*$ and $b^*$ values varied depending on the shade of cement and this tendency was apparent when thickness ratio of ZpexSmile was greater. TP values were not affected by shade of cement and thickness ratio. For flexural strength, the biaxial flexural strength of layered specimens was an intermediate value between Zpex and ZpexSmile; large thickness ratio of ZpexSmile did not decrease the strength.

From these results, the layering method of zirconia with various translucencies using resin cement of different shades can improve color expression while maintaining sufficient flexural strength.

CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

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