Influence of sintering conditions on translucency, biaxial flexural strength, microstructure, and low-temperature degradation of highly translucent dental zirconia

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There is limited research on the influence of different sintering temperatures on the properties of highly translucent zirconia ceramics. This study demonstrated the influence of different sintering temperatures on the translucency, crystallographic structure, biaxial flexural strength, microstructure, and low-temperature degradation (LTD) of three highly translucent zirconia grades, i.e., 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP), 4 mol% yttria-partially-stabilized zirconia (4Y-PSZ), and 5 mol% yttria-PSZ (5Y-PSZ). The specimens were characterized using colorimetry, X-ray diffraction, scanning electron microscopy, Weibull analysis, and LTD tests (134°C; 20 h). The increase in the sintering temperature did not affect the translucency of 3Y-TZP, whereas it increased the translucencies of 4Y-PSZ and 5Y-PSZ. All the zirconia grades exhibited grain enlargement and unchanged biaxial flexural strengths with the increase in the sintering temperature. The degradation of 3Y-TZP and 4Y-PSZ at a sintering temperature of 1,600°C was faster than that at other sintering temperatures.

Keywords: Highly translucent zirconia, Sintering condition, Translucency, Biaxial flexural strength, Weibull analysis

INTRODUCTION

Ceramic materials are widely utilized in dentistry because their aesthetics, chemical durability, and biocompatibility are superior to those of conventional porcelain-fused-to-metal restorations1-8. However, ceramics are brittle, which limits their clinical applications in the posterior regions9. Zirconia-based restorations are presently popular in dentistry owing to their excellent aesthetics and biocompatibility1-4. Furthermore, full-contour zirconia restorations have attracted significant attention in recent times for posterior dental restorations6-10. The 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) are one of the most frequently used materials for restorations owing to their excellent mechanical properties13. However, they are susceptible to low-temperature degradation (LTD) owing to the deterioration in their mechanical strength5,11. This deterioration occurs because the irritation that is caused by mechanical, thermal, and chemical factors increases the t-m (tetragonal-monoclinic) transformation and induces high volume changes of up to 5%. The opaque white appearance of 3Y-TZP is also a critical drawback in esthetic applications2.

Several varieties of highly translucent zirconia have been applied in dentistry. The Al2O3 content in the first highly translucent zirconia ceramic, known as the second generation of zirconia, was lower than that in the conventional 3Y-TZP. The low Al2O3 content increased the translucency of the second generation of zirconia; however, it is also increased the susceptibility of the highly translucent 3Y-TZP to aging. Recently, the latest generation of highly translucent zirconia ceramics, with a higher content of yttria (>=4 mol%) than that in 3Y-TZP, has been introduced12. These ceramics are often referred to as yttria-partially-stabilized zirconia (Y-PSZ), and they are classified based on the yttria content as 4 mol% Y-PSZ (4Y-PSZ) and 5 mol% Y-PSZ (5Y-PSZ). The increase in the yttria content increases the content of the cubic zirconia (c-ZrO2) phase, and the consequent presence of numerous isotropic cubic crystals increases the translucency of the ceramics13,14. However, the high content of the c-ZrO2 phase decreases the strength of 4Y-PSZ and 5Y-PSZ as compared to that of 3Y-TZP14-20.

The increase in the sintering temperature and sintering time increases the susceptibility of the conventional 3Y-TZP to aging25. However, the influence of the different sintering temperatures on the characteristics of the second and latest generation of highly translucent zirconia grades remains unexplored22-24. The present in vitro study demonstrated the influence of the different sintering conditions on the translucency, crystallographic structure, biaxial flexural strength, microstructure, and LTD of three highly translucent zirconia grades i.e., 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. The null hypothesis was that the different sintering conditions did not affect the translucency, crystallographic structure, biaxial flexural strength,

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microstructure, and LTD behavior of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ.

**MATERIALS AND METHODS**

A summary of the experimental setup is shown in Fig. 1. The three highly translucent zirconia grades used in this study were 3Y-TZP (Zpex, Tosoh, Tokyo, Japan), 4Y-PSZ (Zpex 4, Tosoh), and 5Y-PSZ (Zpex Smile, Tosoh). Forty disk-shaped specimens with diameters and thicknesses of 14.5 mm and 1.2 mm, respectively, were prepared for each zirconia grade (Table 1). These specimens were assigned to four subgroups \((n=10)\) according to the sintering temperatures \(i.e., 1,450°C, 1,500°C, 1,550°C,\) and \(1,600°C\). All the specimens were treated in the sintering furnace (Esthemat Sinta 2, Shofu, Kyoto, Japan) with a holding time of 2 h. The disk-shaped fully sintered specimens were prepared \(via\) mechanical polishing using \#325, \#800, and \#1000 SiC papers, in sequence. Finally, all the polished specimens were thermally treated at 1,000°C for 10 min to eliminate any potential residual stress.

**Translucency**

The color and spectral reflectance of all the highly translucent zirconia grades were measured using colorimetry (CR-13; Konica Minolta Sensing, Tokyo, Japan). The specimens were subjected to measurements over a black (CIE \(L^*\)-28.4, \(a^*=-1.1,\) and \(b^*=-0.1\)) and white (CIE \(L^*=94.1,\) \(a^*=-0.4,\) and \(b^*=1.1\)) background. A smears layer of glycerin was utilized as the coupling medium between the zirconia specimens and the black and white background\(^{23}\). The translucency parameter (TP) was calculated using the following equation:

\[
TP=\sqrt{(L_b-L_w)^2+(a_b-a_w)^2+(b_b-b_w)^2}
\]

where the scripts indicate the color coordinates with the black (b) and white (w) backgrounds. The translucency was statistically compared \(via\) the two-way analysis of variance (ANOVA) that was followed by the Tukey post-hoc test \((\alpha=0.05)\).

**Crystallography**

The crystal structures of the three highly translucent zirconia grades were analyzed \(via\) X-ray diffraction (XRD; D8 Advance, Bruker Optik, Ettlingen, Germany) using Cu Kc radiation \((1.5406 \text{ Å})\) at a current and an accelerating voltage of 40 mA and 40 kV, respectively. The top surface of each disk-shaped specimen was analyzed over a 20 range of 20–90° with a step size of 0.02° for 2 s. Rietveld analyses were performed to measure the relative zirconia phase content \((c-ZrO_2, t-ZrO_2, m-ZrO_2)\) and the unit cell dimensions using the software program, TOPAS Academic V5 (Coelho software, Brisbane, Australia). The calculated unit cell parameters were applied to calculate the \(Y_2O_3\) content of the \(t-ZrO_2\) phase in the highly translucent zirconia grades using the methods reported by Krogstad \(et\ al.\)\(^{26}\).

**Biaxial flexural strength test**

The biaxial flexural strength tests were performed using a piston-on-three-ball setup with a universal testing machine (EZ test, Shimadzu, Tokyo, Japan) according

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**Fig. 1** Flow chart detailing the experimental set-up.

**Table 1** Materials investigated in the present study

<table>
<thead>
<tr>
<th>Zirconia grades</th>
<th>Classification</th>
<th>Lot number</th>
<th>(Y_2O_3) Content (wt%)</th>
<th>(Al_2O_3) Content (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zpex</td>
<td>3Y-TZP</td>
<td>ZY308566B</td>
<td>5.34</td>
<td>0.049</td>
</tr>
<tr>
<td>Zpex 4</td>
<td>4Y-PSZ</td>
<td>ZY408625B</td>
<td>6.96</td>
<td>0.048</td>
</tr>
<tr>
<td>Zpex Smile</td>
<td>5Y-PSZ</td>
<td>ZY558557B</td>
<td>9.34</td>
<td>0.047</td>
</tr>
</tbody>
</table>
to the ISO 6872 standard\textsuperscript{27}. A digital caliper (Mitutoyo, Kanagawa, Japan) was utilized to measure the thickness and diameter of the fully sintered specimens before the test. The crosshead speed was maintained at 0.5 mm/min until the occurrence of fracture. The biaxial flexural strength was calculated using the following equation:

\[ S = -0.2387P(X - Y)/d^2 \]

where \( S \) is the biaxial flexural strength (MPa), \( P \) is the fracture load (N), and \( d \) is the thickness (mm) of the specimen disk at the origin of the fracture. \( X \) and \( Y \) were determined as follows:

\[ X = (1 + \nu) \ln(B/C)^2 + [(1 - \nu)/2](B/C)^2 \]
\[ Y = (1 + \nu) \ln(A/C)^2 + [(1 - \nu)/2](A/C)^2 \]

where \( \nu \) is the Poisson’s ratio, \( A \) (mm) is the radius of the support circle, \( B \) (mm) is the radius of the loaded area, and \( C \) (mm) is the radius of the specimen. The Poisson’s ratio was set to 0.3, as in a previous study\textsuperscript{28}).

The results of the biaxial flexural strength tests were statistically compared using the Weibull analysis. The Weibull parameters were calculated \textit{via} maximum-likelihood estimation, and the likelihood ratio was employed to calculate the confidence interval bounds. A likelihood contour method was applied to determine the statistical differences between the compared Weibull distributions. There were no significant differences in the intersected contour plots that were obtained using the likelihood contour method, as previously described by Thompson\textsuperscript{29}). The analysis was performed at a significance level of \( \alpha = 0.05 \) using the software packages, R3.6.1 and weibullR (R Foundation for Statistical Computing).

\textbf{Microstructural analysis}

One specimen of each zirconia grade after the biaxial flexural strength test was ultrasonically cleaned with acetone for 3 min, dried under oil-free air pressure, and placed in vacuum overnight. The specimens were subjected to thermal etching in the sintering furnace (Rapid High Temperature Furnace, Bulten Kanthal, Hallstahammar, Sweden) at 1,200°C for 10 min, assuming a grain boundary network. A thin layer of platinum was coated on the specimens \textit{via} ion sputtering (E102, Hitachi High-Technologies, Tokyo, Japan). Subsequently, the specimens were subjected to morphological analyses \textit{via} scanning electron microscopy (SEM; S-4500, Hitachi High-Technologies) with an acceleration voltage of 15 kV, an emission current of 10 µA, and a working distance of 15 mm. We employed magnifications of 20,000× and 5,000× for the microstructural analyses and the fracture surface analyses, respectively. The average grain size for each specimen of the highly translucent zirconia grades was measured \textit{via} the linear intercept method using at least 500 grains. Here, the number of interceptions that were made by a test line of known length was counted using the ImageJ software (National Institutes of Health, Bethesda, MD, USA)\textsuperscript{30}).

\textbf{LTD test}

The LTD tests were performed according to the ISO 13356 standard. The specimens of each highly translucent zirconia grade were placed in an autoclave (ETTAS 300B, AS ONE, Osaka, Japan) at 134°C under 2 bar for up to 20 h (\( n = 3 \)/experimental group). The volume fraction of the m-ZrO\(_2\) phase in the specimens after the treatment in the autoclave was determined using XRD (D8 Advance, Bruker Optik). The top surface of each specimen was analyzed over a 20 range of 27–33° with a step size of 0.02° for 2 s. The increase in the volume fraction of m-ZrO\(_2\) represented the difference in the m-ZrO\(_2\) content of a degraded surface and that of a polished surface before autoclaving. The volume fraction of m-ZrO\(_2\) was calculated using the equation reported by Toraya \textit{et al}\textsuperscript{31}).

\section*{RESULTS}

\textbf{Translucency}

The results of the translucency measurements are presented in Fig. 2. The TP of the highly translucent zirconia grades ranged from 19.07±0.47 for Zpex at 1,500°C to 26.44±0.73 for Zpex Smile at 1,550°C. The translucency of Zpex (3Y-TZP) was significantly lower than that of Zpex 4 and Zpex Smile. The TP of Zpex remained unaffected with the increase in the sintering temperature, whereas the TPs of Zpex 4 and Zpex Smile increased with the increase in the sintering temperature.

\textbf{Crystallography}

The results of the Rietveld analyses are summarized in Table 2. The content of the t-ZrO\(_2\) phase in the specimens ranged from 19.07±0.47 for Zpex at 1,500°C to 26.44±0.73 for Zpex Smile at 1,550°C. The content of the t-ZrO\(_2\) phase in Zpex 4 was 62–66 wt%, which was intermediate between the contents of
Table 2  Summary of the results of the Rietveld analysis and the calculated Y₂O₃ content in the tetragonal phase (mol%)

<table>
<thead>
<tr>
<th>Zirconia grades</th>
<th>Sintering conditions (°C)</th>
<th>Content (wt%) of the different ZrO₂ phases</th>
<th>Y₂O₃ content in the tetragonal phase (mol %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>tetragonal</td>
<td>cubic</td>
</tr>
<tr>
<td>Zpex</td>
<td>1,450</td>
<td>86.00</td>
<td>13.85</td>
</tr>
<tr>
<td></td>
<td>1,500</td>
<td>82.50</td>
<td>17.36</td>
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<tr>
<td></td>
<td>1,550</td>
<td>81.89</td>
<td>17.89</td>
</tr>
<tr>
<td></td>
<td>1,600</td>
<td>79.29</td>
<td>20.70</td>
</tr>
<tr>
<td>Zpex 3Y-TZP</td>
<td>1,450</td>
<td>66.33</td>
<td>33.49</td>
</tr>
<tr>
<td></td>
<td>1,500</td>
<td>63.98</td>
<td>35.77</td>
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<tr>
<td></td>
<td>1,550</td>
<td>62.83</td>
<td>37.06</td>
</tr>
<tr>
<td></td>
<td>1,600</td>
<td>63.71</td>
<td>36.24</td>
</tr>
<tr>
<td>Zpex 4Y-PSZ</td>
<td>1,450</td>
<td>43.27</td>
<td>56.48</td>
</tr>
<tr>
<td></td>
<td>1,500</td>
<td>43.24</td>
<td>56.71</td>
</tr>
<tr>
<td></td>
<td>1,550</td>
<td>48.17</td>
<td>51.56</td>
</tr>
<tr>
<td></td>
<td>1,600</td>
<td>44.36</td>
<td>54.80</td>
</tr>
</tbody>
</table>

Fig. 3  Summary of the Weibull analysis for the biaxial flexural strengths of Zpex (3Y-TZP), Zpex 4 (4Y-PSZ), and Zpex Smile (5Y-PSZ) under different sintering conditions.  Weibull plot with 95% confidence bands: (a) Zpex, (c) Zpex 4, and (e) Zpex Smile. The horizontal dotted line indicates the Weibull characteristic strength. (Probability of failure=63.2%). Weibull contour plots for (b) Zpex, (d) Zpex 4, and (f) Zpex Smile. The intersected contour plots indicate no significant statistical difference.
Table 3 Summary of the Weibull analysis

<table>
<thead>
<tr>
<th>Zirconia Grades</th>
<th>Sintering Conditions (°C)</th>
<th>n</th>
<th>Weibull Modulus (m)</th>
<th>95% Confidence level of Weibull Modulus (m)</th>
<th>Characteristic Strength σ₀ (B63.2) in MPa</th>
<th>95% Confidence level at σ₀ (B63.2) in MPa*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zpex</td>
<td>1,450</td>
<td>10</td>
<td>8.4</td>
<td>4.8–13.6</td>
<td>1,259</td>
<td>1,160–1,359 (bc)</td>
</tr>
<tr>
<td></td>
<td>1,500</td>
<td>10</td>
<td>7.8</td>
<td>4.5–12.9</td>
<td>1,348</td>
<td>1,237–1,466 (abc)</td>
</tr>
<tr>
<td></td>
<td>1,550</td>
<td>10</td>
<td>4.1</td>
<td>2.4–6.8</td>
<td>1,194</td>
<td>1,016–1,390 (c)</td>
</tr>
<tr>
<td></td>
<td>1,600</td>
<td>10</td>
<td>7.2</td>
<td>4.0–12.3</td>
<td>1,356</td>
<td>1,233–1,485 (abc)</td>
</tr>
<tr>
<td>Zpex 4</td>
<td>1,450</td>
<td>10</td>
<td>10.1</td>
<td>5.9–16.3</td>
<td>1,410</td>
<td>1,324–1,507 (ab)</td>
</tr>
<tr>
<td>4Y-PSZ</td>
<td>1,500</td>
<td>10</td>
<td>12.4</td>
<td>7.7–19.4</td>
<td>1,396</td>
<td>1,325–1,468 (ab)</td>
</tr>
<tr>
<td></td>
<td>1,550</td>
<td>10</td>
<td>9.0</td>
<td>4.8–15.2</td>
<td>1,376</td>
<td>1,270–1,478 (ab)</td>
</tr>
<tr>
<td></td>
<td>1,600</td>
<td>10</td>
<td>19.4</td>
<td>11.3–30.7</td>
<td>1,382</td>
<td>1,338–1,431 (ab)</td>
</tr>
<tr>
<td>Zpex Smile</td>
<td>1,450</td>
<td>10</td>
<td>6.2</td>
<td>3.5–10.3</td>
<td>968</td>
<td>867–1,076 (d)</td>
</tr>
<tr>
<td>5Y-PSZ</td>
<td>1,500</td>
<td>10</td>
<td>9.6</td>
<td>5.4–16.1</td>
<td>1,026</td>
<td>955–1,096 (d)</td>
</tr>
<tr>
<td></td>
<td>1,550</td>
<td>10</td>
<td>11.7</td>
<td>6.5–18.9</td>
<td>988</td>
<td>929–1,043 (d)</td>
</tr>
<tr>
<td></td>
<td>1,600</td>
<td>10</td>
<td>6.9</td>
<td>4.0–11.6</td>
<td>906</td>
<td>824–994 (d)</td>
</tr>
</tbody>
</table>

*Different letters indicate the statistical difference.

the t-ZrO₂ phase in Zpex and Zpex Smile. The increase in the sintering temperature lowered the content of the t-ZrO₂ phase in Zpex and Zpex 4, whereas it did not affect the content of the t-ZrO₂ phase in Zpex Smile. Furthermore, the increase in the sintering temperature lowered the Y₂O₃ content of the t-ZrO₂ phase in Zpex and Zpex 4 (Table 2).

**Biaxial flexural strength test**
A summary of the Weibull analyses for the biaxial flexural strength tests is presented in Fig. 3 and Table 3. The results revealed that the biaxial flexural strength of Zpex Smile was significantly lower than that of Zpex and Zpex 4. The biaxial flexural strengths of Zpex and Zpex 4 were comparable. The intersection of all the Weibull contour plots for each zirconia grade indicated that the sintering temperature did not affect the biaxial flexural strengths of the three highly translucent zirconia grades. The lowest Weibull modulus (m=4.1) among all the groups was presented by Zpex at 1,550°C, while the highest Weibull modulus (m=19.4) among all the groups was presented by Zpex 4 at 1,600°C.

**Microstructural analysis**
The results of the microstructural analyses via SEM, which revealed the grain size distribution with the average grain size, are presented in Fig. 4. The grain size of zirconia increased with the increase in the sintering temperature. Zpex exhibited uniform grains of the lowest size, whereas Zpex Smile presented a substantial number of large grains at all sintering temperatures. Moreover, Zpex Smile presented a bimodal grain size distribution.

The ultrastructural images that were obtained from the fractography analysis are presented in Fig. 5. Zpex presented an intergranular fracture at all sintering temperatures. The dominant fracture mode for Zpex 4 was an intergranular fracture, whereas Zpex Smile presented a completely transgranular fracture at all sintering temperatures.

**LTD test**
The results of the LTD test that indicated the increase in the content of the m-ZrO₂ phase are summarized in Fig. 6. The Zpex specimens that were sintered at 1,550 and 1,600°C exhibited degradation after 5 h of LTD aging, while demonstrating comparable aging behaviors at both sintering temperatures. When Zpex was sintered at 1,450°C, degradation did not occur even after 20 h of LTD aging. After sintering at all temperatures, Zpex 4 did not exhibit degradation even up to 10 h of LTD aging. After 10 h of LTD aging, the Zpex 4 specimen
Fig. 5  Results of fractography analysis of Zpex (3Y-TZP), Zpex 4 (4Y-PSZ), and Zpex Smile (5Y-PSZ) under different sintering conditions. Zpex (3Y-TZP) presents an intergranular fracture at all sintering temperatures. Zpex 4 (4Y-PSZ) that is sintered at 1,450 and 1,500°C presents an intergranular fracture, while Zpex 4 (4Y-PSZ) that is sintered at 1,550 and 1,600°C presents a transgranular fracture. Zpex Smile (5Y-PSZ) presents a transgranular fracture at all sintering temperatures.

that was sintered at 1,600°C started to degrade; after 20 h, its \( m - \text{ZrO}_2 \) volume fraction had reached 12.9%. A limited amount of \( m - \text{ZrO}_2 \) was detected in the Zpex Smile specimen even after 20 h of LTD aging, while its aging behavior remained unchanged at all sintering temperatures.

DICUSSION

The present study revealed that the different sintering temperatures did not affect the translucency of Zpex, the biaxial flexural strength of all the zirconia grades, and the aging behavior of Zpex Smile. However, the variation in the sintering temperature exerted a substantial influence on the translucency of Zpex 4 and Zpex Smile; furthermore, it also affected the microstructure of all the zirconia grades. The increase in the sintering temperature affected the crystallographic structure and the aging behavior of Zpex and Zpex 4. Therefore, the null hypothesis, which stated that the different sintering conditions did not affect the translucency, crystallographic structure, biaxial flexural strength, microstructure, and LTD of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ, was partially rejected. To the best of our knowledge, no other study has systematically assessed the influence of the different sintering temperatures on the translucency, crystallographic structure, mechanical properties, and aging behavior of highly translucent zirconia grades.

The increase in the sintering temperature did not affect the TP of Zpex, whereas it increased the TPs of Zpex 4 and Zpex Smile. Zpex presented a low translucency owing to the presence of numerous grain boundaries; furthermore, the remaining \( \text{Al}_2\text{O}_3 \) grains were diffused or repositioned near the grain boundaries, thereby facilitating the scattering of light between them\(^{20} \). Generally, materials with large grains exhibit high translucency owing to the limited number
of grain boundaries; however, these materials are highly susceptible to not only transformation but also a deterioration in their mechanical strength. The average grain sizes of some of the Zpex and Zpex 4 specimens in the present study were comparable. However, the TPs of these specimens were significantly different owing to the difference in the c-ZrO$_2$ content because the effect of the c-ZrO$_2$ content on the translucency was greater than that of the grain size. There has been extensive research on the effects of the sintering temperature on the translucency of zirconia. The previous research revealed that the increase in the sintering temperature increased the translucency and grain size; however, it decreased the porosity and induced the densification of the crystal structure of zirconia. However, there were no significant changes in the TP of Zpex with the increase in the sintering temperature in the present study. Zpex presented large grains at 1,600°C without any significant change in its translucency. This was attributed to the effect of the microstructure and composition on the optical properties of the material. The microstructure of the Zpex specimen was analyzed using the Rayleigh scattering model, where all grains were assumed to have identical diameters and follow an orbital distribution. In our experiments, the Zpex specimen exhibited altered grain sizes at different sintering temperatures, along with a unimodal grain size distribution. This phenomenon suggested the manifestation of birefringence on the tetragonal (but not cubic) phases in Zpex, which resulted in increased light scattering. The translucency of Zpex 4 and Zpex Smile increased with the increase in the sintering temperature owing to the decrease in the intercept area between the grain boundaries. The results of the present study were similar to those of the study by Nazmiye et al., where no significant differences in the TP of 3Y-TZP were reported at different sintering temperatures (1,450–1,600°C). Stawarczyk et al. investigated the impact of different sintering temperatures on the flexural strength, contrast ratio, and grain size of Y-TZP. They reported the increase in the contrast ratio and grain size with the increase in the sintering temperature. Recent studies reported that the translucency and flexural strength of 5Y-PSZ were unaffected by the change in the sintering temperature; however, the grain size increased with the increase in the sintering temperature.

The presence of 10 wt% of Y$_2$O$_3$ increased the content of the c-ZrO$_2$ phase in Zpex Smile, thereby increasing the translucency of Zpex Smile. However, there was a deterioration in the mechanical properties of the material. Therefore, the biaxial flexural strength of Zpex Smile was significantly lower than that of Zpex and Zpex 4. On the other hand, the biaxial flexural strength of Zpex 4 was comparable to that of Zpex, owing to their similar grain size distributions and fracture patterns, combined with the above 60% t-ZrO$_2$ phase content in the former. Zirconia ceramics that showed high biaxial flexural strengths exhibited intergranular patterns on their fracture surfaces, which was in agreement with an earlier report by Zhang et al. According to the results of the Rietveld analysis, when the sintering temperature increased, the Y$_2$O$_3$ contents in the t-ZrO$_2$ phases from the Zpex and Zpex 4 specimens decreased from 2.64 to 2.49 and from 2.83 to 2.67, respectively. The decreased Y$_2$O$_3$ contents resulted in higher susceptibility to LTD, indicating that the content of the tetragonal phase of Y$_2$O$_3$ in zirconia ceramics can serve as a predicting factor of their LTD behaviors. This observation could be further verified by the higher Y$_2$O$_3$ content in Zpex Smile as compared with those in the other two ceramics, which enhanced its resistance to LTD at all sintering temperatures.

The sintering temperature did not affect the biaxial flexural strength of the three highly translucent zirconia grades in the present study. These results were consistent with those of the previous studies that indicated no significant changes in the biaxial flexural strength of zirconia with the increase in the sintering temperature. They reported that the sintering temperature did not affect the biaxial flexural strength of Y-TZP. Furthermore, recent studies reported that the increase in the sintering temperature from 1,450 to 1,600°C did not affect the flexural strength of 5Y-PSZ. Nazmiye et al. assessed the flexural strengths of 3Y-TZP and 5Y-PSZ that were sintered at 1,350–1,600°C. The results revealed that the flexural strengths at 1,350°C were significantly lower than those at 1,450 and 1,600°C. Stawarczyk et al. reported that Y-TZP presented comparable biaxial flexural strengths in the sintering temperature range of 1,400–1,550°C; however, the biaxial flexural strength decreased significantly at the temperatures above and below this range. Therefore, the results obtained by Stawarczyk et al. and Nazmiye et al. were consistent. It was concluded that the optimal sintering temperature range to maintain a high flexural strength was 1,450–1,600°C.

The microstructural analysis revealed the presence of extremely large grains in Zpex that was sintered at 1,600°C and Zpex 4 that was sintered at 1,550°C or above. The increase in the sintering temperature induced the enlargement of grains in all the zirconia grades in the present study. Inokoshi et al. investigated the influence of the sintering conditions on the properties of 3Y-TZP. They reported that 3Y-TZP presented a bimodal grain size distribution at a sintering temperature of 1,500°C or above, which was consistent with the results of the present study. Manière et al. reported the rapid increase in the grain size of Zpex Smile at a sintering temperature of 1,550°C or above. They also revealed that the slow grain growth at the sintering temperatures below 1,550°C exerted a significant influence on the intragranular porosity.

The fractography analysis revealed that Zpex and Zpex Smile exhibited completely intergranular and transgranular fractures, respectively. The dominant fracture mode in Zpex 4 was an intergranular fracture. It is known that the zirconia grades with high flexural strength tend to exhibit intergranular fractures. The results of the fractography analysis in the present study were consistent with the results reported by Zhang.
et al.\textsuperscript{38})

The LTD tests were conducted for up to 20 h using an autoclave in the present study. Zhang et al.\textsuperscript{43} reported that 1 h of aging at 134°C corresponds to 1–2 years of aging at 37°C. This report indicated that the LTD tests in the present study corresponded to 20–40 years of aging at the temperature of the body (37°C). The results of the LTD tests revealed that all the zirconia grades were resistant to aging for up to 5 h. The Zpex specimens that were sintered at 1,550 and 1,600°C started to degrade after 5 h of aging, whereas the Zpex 4 specimens sintered at 1,600°C exhibited degradation after 10 h of aging. The aging resistance of Zpex at a sintering temperature of 1,500°C or below and Zpex 4 at a sintering temperature of 1,550°C or below was attributed to the influence of the surface polishing. Despite the elimination of the compressive surface stress via heat treatment, the surface polishing induced ferroelastic domain switching that increased the aging resistance of Zpex and Zpex \textsuperscript{44,44}. The increase in the aging resistance of surface-treated zirconia was reported in previous studies\textsuperscript{45-50}. Muñoz et al.\textsuperscript{45} reported that mechanical grinding induced the development of fine zirconia grains layer (10–20 nm) immediately underneath the zirconia surface\textsuperscript{46}). The presence of these fine zirconia grains, a deformed zone, and a tetragonal-to-monoclinic transformed zone contributed to the high aging resistance of Zpex and Zpex 4. Inokoshi et al.\textsuperscript{21} reported that the optimal sintering temperature for 3Y-TZP ceramics to ensure a high aging resistance was 1,450°C, which was consistent with the results of the present study. Zpex Smile was not degraded at any sintering temperature in the present study. This was consistent with the observations in previous studies, where Zpex Smile exhibited the highest resistance to LTD\textsuperscript{16,18,24,38}). The results of Rietveld refinement revealed that the Y\textsubscript{2}O\textsubscript{3} content in the t-ZrO\textsubscript{2} phase of Zpex Smile was more than 3.8 wt%, which lowered the transformability of the t-ZrO\textsubscript{2} phase. Moreover, Zpex Smile specimen also demonstrated a very high c-ZrO\textsubscript{2} phase content (>50%), which improved its aging resistance in a humid environment. As such, this specimen exhibited no signs of degradation at all the sintering temperatures applied here\textsuperscript{18,50}). Some previous studies reported the high susceptibility of large zirconia grains to LTD\textsuperscript{21,50}, and the LTD behavior of Zpex and Zpex 4 in the present study were consistent with this report. The recommended sintering temperature for Zpex, Zpex 4, and Zpex Smile to ensure a high aging resistance was concluded to be 1,500°C or below, 1,550°C or below, and 1,600°C or below, respectively.

We investigated the influence of different sintering temperatures on the basic properties of highly translucent zirconia grades in the present study. One limitation of this study was that sintering time for 2 h was the only parameter thoroughly investigated, therefore future work in this field should explore the impact of other sintering parameters, such as a shortened sintering time, on the properties of highly translucent zirconia grades.

CONCLUSIONS

The present study demonstrated the effects of different sintering temperatures on the translucency, zirconia phase content, grain size, and LTD resistance behavior of three highly translucent zirconia ceramics, i.e., 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. Our principal conclusions were as follows:

1. The recommended sintering temperatures to achieve a higher aging resistance in 3Y-TZP were 1,450 and 1,500°C.
2. When sintered at 1,500°C (or below), 4Y-PSZ exhibited enhanced translucency and improved aging resistance, as compared with those of 3Y-TZP. On the other hand, the biaxial flexural strengths of both samples at this sintering temperature, were comparable.
3. The optimal sintering temperatures in order to achieve a high translucency, satisfactory biaxial flexural strength, and enhanced aging resistance in 5Y-PSZ, were determined to be 1,550 and 1,600°C.

As a closing note, it is necessary to continue the work in this subject field by exploring the impact of other sintering parameters, such as the sintering time (sintering speed), on the properties of highly translucent zirconia ceramics.

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REFERENCES

11) Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness, and microstructure of a selection of all-


