Sintering mode of a translucent Y-TZP: Effects on its biaxial flexure fatigue strength, surface morphology and translucency

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Abstract

Objective: This investigation evaluated the effect of two sintering modes of a translucent zirconia (Y-TZP) on its surface roughness, topography, phase-transformation ($t \rightarrow m$), translucency and biaxial flexure fatigue strength.

Materials and Methods: To do so, 50 Y-TZP discs ($\phi = 15$ mm; thickness = 1.2 mm; IPS e.max ZirCAD LT) were prepared and divided into two groups: Standard mode (SM) and Fast mode (FM). Staircase fatigue testing was performed (piston-on-three balls set-up, ISO 6872:2015), as well as surface roughness, profilometry, scanning electron microscopy (SEM-FEG), energy dispersive X-ray spectroscopy (EDX), phase transformation ($t \rightarrow m$) using X-ray diffraction analysis (XRD), translucency parameter analysis (TP and TP00) and fractography.

Results: The results showed no statistical significant differences for roughness parameters ($p > 0.05$, SM: $Ra = 0.13 \pm 0.02$, $Rz = 1.21 \pm 0.26$ and $RSm = 24.91 \pm 2.19$; FM: $Ra = 0.14 \pm 0.03$, $Rz = 1.32 \pm 0.25$ and $RSm = 24.68 \pm 2.16$) or flexural fatigue strength (SM: 512 (464–560) MPa; FM: 542 (472–611) MPa) between the groups. In addition, similarity in surface morphological features (SEM and profilometry), composition and phases (EDX and XRD) was observed between the firing protocols. Fractography showed that the failure origin occurred on the tensile side. Sintering mode did not affect the TP ($F = 0.001$, $p = 0.97$) and TP00 ($F = 0.12$, $p = 0.72$).

Conclusions: Therefore, the fast-sintering mode is suggested as a viable alternative to the standard mode since it does not influence the evaluated surface morphology, microstructure, fatigue strength and translucency of a translucent monolithic zirconia.

Clinical Significance: The fast sintering mode is a viable alternative for zirconia without compromising its topography, microstructure, mechanical performance or translucency.

Keywords
fatigue, fractography, prosthodontics, sintering, translucent zirconia
1 | INTRODUCTION

The consolidation of metal-free ceramics in dental practice has led to a breakthrough in this segment, from glass ceramics ideally used as a veneering material, to reinforced ceramics such as those based on aluminium or zirconium oxide developed for manufacturing crown and bridge infrastructures. With highly advantageous properties, zirconia ceramics are increasingly being used in fabricating infrastructures as well as monolithic restorations. Zirconia is susceptible to phase transformation as it is a polymorphic material and has three main phases (monoclinic, tetragonal and cubic). The interest and ability to use zirconia in monolithic restorations has recently been increasing, especially due to the development of new generations of this material which have a higher translucency level that benefits its esthetics, as well as the increased knowledge in working the material microstructure to obtain such property.

Advancement and technological speed have led to faster development and innovation of different materials, and this is not different in the dental field. Additionally, several types of ceramic systems have been developed and introduced to the market in order to meet the high expectations of both patients and dentists for natural, biocompatible and long-lasting restorations. The role of zirconia ceramics in this context has been highlighted and new possibilities for esthetics, strength and technical simplification have been attracting the dental community, especially the group of CAD/CAM professionals with a one-day dental care concept. The main cause of failure for these materials in ceramic restorations remain related to flaws and stresses located on the surface where the tensile stresses are concentrated.

Along with the evolution of zirconia ceramic generations, the sintering process of this material has been acquiring prominence and development and innovation of different materials, and this is not different. The firing process reduces pores between grains and increases zirconia's final density, thus producing less light scattering and more light transmission. In addition, prolonged sintering time has been reported as being capable to increase the flexural strength of a translucent monolithic zirconia as a function of grain size variation and phase transformation (t → m). The influence of this firing has caused interest in the scientific community following the introduction of short sintering cycles. Thus, many investigations have studied the effects of changes on sintering time, temperature, translucency, grain size and biaxial flexural strength of the zirconia infrastructure. However, the effect of these changes on monolithic zirconia's properties remains debatable.

Given these aspects, it seems relevant to accordingly analyze the effect of different sintering processes on the surface morphology, phase transformation, fatigue strength, fractographic features and translucency of a novel translucent monolithic zirconia. Therefore, the hypotheses of this study were that the Fast sintering mode would negatively influence the: 1) Morphology and microstructure; 2) Mechanical strength; and 3) Translucency parameter of the evaluated zirconia compared to the Standard sintering mode.

2 | MATERIAL AND METHODS

2.1 | Specimen preparation

Pre-sintered ceramic blocks of 3Y-TZP zirconia (Batch number W03411, IPS e.max ZirCAD LT, Ivoclar Vivadent AG, Schaan, Liechtenstein) were ground with #600 to #1200 silicon carbide (SiC) papers (3M ESPE, St Paul, USA) under water cooling to obtain ceramic cylinders which were then sectioned into discs as recommended by International Organization for Standardization (ISO) 6872:2015. These discs were obtained with a diamond saw machine (Isomet® 1000, Precision Sectioning Saw, Buehler, Lake Bluff, Illinois, USA) under constant water cooling, in dimensions of 18 mm (Ø) and 1.6 mm (thickness). All discs were polished with #1200 grit Sic followed by an ultrasonic bath with isopropyl alcohol 78% for 5 min to remove the irregularities caused by the cylinder cutting process, and then sintered to obtain the final dimensions of 15 mm (Ø) and 1.2 mm (thickness).

The discs were randomly distributed into two groups for the sintering process:

- **Standard mode group** (SM): the standard 57-min zirconia sintering program was used in a Programat® CS4 furnace (Ivoclar Vivadent AG, Schaan, Liechtenstein) (n = 25).
- **Fast mode group** (FM): the program for fast sintering (37 min) was used in the same furnace (n = 25), according to the manufacturer's instructions. All discs were carefully measured after sintering, and discs with discrepancies in size above the recommended deviation (1.2 ± 0.2 mm) as indicated by ISO 6872:2015 were used for surface analysis.

2.2 | Surface topography characterization

2.2.1 | Surface roughness

The discs (n = 20, N = 40) were analyzed for surface roughness to verify the surface morphology, with the following three parameters being verified: Ra (mean roughness), Rz (average distance between the five highest peaks and five deepest valleys) and RSm (spacing parameter) through three parallel and equidistant readings (3 mm) with a scanning speed of 0.8 mm/s following the ISO 4287:1997 (ISO, 1997), with a Gaussian filter and a cut-off value of 0.8 mm using a roughness meter (Surftest SJ 400, Mitutoyo, Tokyo, Japan).

2.2.2 | Profilometry

Three readings per specimen (n = 3, N = 6) were performed for qualitative analysis of the two-dimensional and three-dimensional surface
using an optical profilometer (Wyko NT1100, Veeco, Tucson, USA). Surface topography analysis was carried out using the Wyko Vision 32 software program (Veeco, Tucson, USA) with 20× magnification over an area of 301.3 x 229.2 µm.

2.2.3 | Scanning electron microscopy—Field emission gun (SEM-FEG) and energy dispersive X-ray spectroscopy (EDX)

EDX analysis was performed to see if the different sintering modes would promote any modification in the specimens’ surface chemical structure (n = 2, N = 4). An energy dispersive X-ray spectrometer coupled to SEM (Inspect S50, FEI Company, Brno, Czech Republic) was used for the chemical analysis. High resolution field emission scanning electron micrographs (SEM-FEG: 15 and 30 kV; Magellan 400L, FEI Company, Brno–Moravia, Czech Republic) at 40,000×, 80,000×, and 200,000× magnification were taken to morphologically characterize the specimen surfaces (n = 2, N = 4).

2.2.4 | X-ray diffraction analysis (XRD)

XRD analysis (n = 2, N = 4) for phase transformation inspection was performed with CuK radiation (Grazing Incidence X-ray Diffraction; Philips X’pert PRO MRD, Almeio, Netherlands; Cu wavelength = 0.15418 nm; range (—2) between 10° C and 100° C; 2° min—1 speed; 40 kV; 20 mA (Philips PW 1830/1840)). The crystalline phases were identified after comparing the experimental spectra with standard diffraction spectra from the Joint Committee on Powder Diffraction Standards (JCPDS) and ICSD (Inorganic Crystal Structure) databases. A program called HighScore performed the spectrum assignments.

2.3 | Translucency parameter

A spectrophotometer with an integrated sphere (CM-2600d, Konica Minolta, Osaka, Japan) was used to perform the translucency measurements. Color and spectral distribution were measured according to the International Commission on Illumination (CIE)\L’*a*b* system (Spectra Magic NX software, Konica Minolta, Osaka, Japan). The standard illuminant D65 was set in reflectance mode with ultraviolet inclusion, observer angle 2° and specular reflection included.\textsuperscript{27} The spectrophotometer was calibrated to the black and white pattern prior to measuring, as provided by the manufacturer. The background color coordinates were white (L: 84.95; a: —0.38; b: 2.93) and black (L: 25.58; a: —0.15; b: —0.24). An optical contact between the specimen and the background was used.

The translucency parameter was evaluated according to the color difference between the black and white backgrounds measurement (n = 13, N = 26). This difference was calculated using both CIE\L’*a*b* (TP) and CIEDE2000 (TP\textsubscript{00}), respectively, Equations (1) and (2)\textsuperscript{28,29}:

\[
TP = 
-2 \times \left( \left( L_b - L_w \right)^2 + \left( a_b - a_w \right)^2 + \left( b_b - b_w \right)^2 \right)^{1/2}.
\]

\[
TP_{00} = \left[ \frac{(L_b - L_w)^2}{K_C S_L} + \frac{(C_b - C_w)^2}{K_C S_C} + \frac{(H_b - H_w)^2}{K_C S_H} \right]^{1/2}.
\]

In which: L is lightness, C is chroma, H is hue, RT is the interaction between C and H differences (blue region); S\textsubscript{c}, S\textsubscript{C}, and S\textsubscript{h} adjust the total color difference for variation in the location of the color difference specimen over white (w) and black (ß) backgrounds in L*, a*, and b* coordinates; K\textsubscript{c}, K\textsubscript{C}, and K\textsubscript{H} are correction terms.\textsuperscript{29}

2.4 | Staircase fatigue test

Prior to the Staircase test, the initial load and the step size were determined based on the results of the monotonic tests (n = 5, N = 10) (50 kgf and 1 mm/min until fracture; DL1000,EMIC São José dos Pinhais, Brazil). An adhesive tape was attached to the compression side of each disc to prevent fragment loss and to provide better contact between the piston and the sample. Flexural strength was calculated according to ISO 6872:2015\textsuperscript{17} using Equations (3) and (4):

\[
X = (1 + \nu) \ln \left( \frac{r_2}{r_3^2} \right)^2 + \left( \frac{1 - \nu}{2} \right) \left( \frac{r_1}{r_3} \right)^2,
\]

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

In which: \nu is the Poisson ratio (0.3),\textsuperscript{25} r\textsubscript{1} is the support circle radius (5.77 mm), r\textsubscript{2} is the loaded area radius (0.8 mm), and r\textsubscript{3} corresponds to the specimen radius (7.5 mm).

The mean monotonic load for fracture (MPa) obtained for each group determined the load profile during the fatigue test. The biaxial flexure fatigue strength (n = 25, N = 50) was obtained based on the specimen survival under the determined load during 20,000 cycles (each loading step). The load was applied with an amplitude ranging from a minimum of 10 MPa to prevent specimen movement at a frequency of 20 Hz.\textsuperscript{24} In addition, 70% of the mean value was assumed as the initial load. If the specimen survived the 20,000 cycles, the

| TABLE 1 | Mean roughness values and standard deviations (µm) according to the sintering mode and the roughness parameter evaluated |
|----------|-------------------------------------------------------------|
| Sintering mode | Ra | Rz | RSm |
| Standard (SM) | 0.13 (0.02) | 1.21 (0.26) | 24.91 (2.19) |
| Fast (FM)    | 0.14 (0.03) | 1.32 (0.25) | 24.68 (2.16) |
FIGURE 1  2D and 3D profilometry images. (A–B) SM; (C–D) FM, respectively.

FIGURE 2  Micrographs (FEG-SEM) 40K×, 80K×, and 2000K× of the grain microstructure of the samples sintered through the standard mode (SM) (A,B,C), fast mode (FM) (D,E,F).
subsequent specimen was cycled at a higher load (±5% of maximum fracture strength). In the case of failure of the tested specimen, the next specimen was cycled at a lower load (−5% of maximum fracture strength). This process was repeated until all tests were completed.

2.4.1 | Fractographic analysis

All fractured specimens were inspected under a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, Germany) for failure origin analysis. Representative specimens were then submitted to SEM (80× and 150× of magnification; Inspect S50, FEI Company, Brno, Czech Republic) to determine failure origin and fractographic characteristics.26

2.5 | Statistical analysis

After the data normality evaluation using the Ryan-Joiner test, roughness, fatigue limit and translucency parameters measurements were evaluated by one-way analysis of variance (ANOVA) and Tukey’s test (p = 5%). Fatigue strength data was calculated24 and the SEM and profilometry images were qualitatively evaluated.

### TABLE 2  Mass composition of specimens by EDX analysis sintered through standard (SM) and fast (FM) modes

| Chemical element | SM        | FM        |
|------------------|-----------|-----------|
| Zirconium        | 69.88%    | 71.31%    |
| Oxygen           | 22.95%    | 23.05%    |

### TABLE 3  Mean values and standard deviation (SD) for ΔL*, Δa*, Δb* and translucency parameters (TP and TP00) of the specimens sintered by the standard (SM) and fast (FM) modes

| Sintering mode | ΔL*        | Δa*        | Δb*        | TP          | TP00       |
|----------------|------------|------------|------------|-------------|------------|
| SM             | −6.7 (1.6) | −1.6 (0.3) | −7.6 (1.7) | 10.3 (2.3)  | 7.4 (1.7)  |
| FM             | −6.6 (0.7) | −1.9 (0.2) | −7.9 (0.6) | 10.5 (0.9)  | 7.6 (0.7)  |

3 | RESULTS

3.1 | Surface topography

Surface roughness was not influenced by the different sintering modes and no differences were observed among the groups for Ra (F = 1.09, p = 0.30), Rz (F = 1.71, p = 0.19), and RSm (F = 0.11, p = 0.74) roughness parameters. Mean roughness values (μm) and standard deviations for all considered parameters are described in Table 1. This data is corroborated by the 2D and 3D profilometry images. Figure 1 shows the peaks in red and the valleys in green.

SEM micrographs show similarity among the specimens (Figure 2). The microstructure of the specimens sintered using the fast mode were visually more homogenous through higher magnification. The EDX results showed almost identical chemical elements of the samples and the mass composition of specimens detected by the analysis are described in Table 2.

XRD graphs are shown in Figure 3, and the peaks exactly represent the zirconia tetragonal phase, regardless of the sintering mode.

3.2 | Translucency parameter

One-way ANOVA showed that the sintering modes employed did not affect the material TP (F = 0.001, p = 0.97) and TP00 (F = 0.12, p = 0.72). Table 3 summarizes the mean and standard deviations for each color coordinate (ΔL*, Δa*, and Δb*) and translucency parameters.
3.3 | Staircase fatigue results and failure analysis

The two groups presented no statistically significant difference (Table 4 and Figure 4). After the analysis of all specimens at different magnitudes, the failures in both groups similarly started from the tensile side. It is possible to observe similar fractographic patterns between the tested groups in Figure 5.

| Sintering mode | Monotonic (SD) | Fatigue strength* |
|----------------|---------------|-------------------|
| SM             | 784 (145)     | 512 (464–560)     |
| FM             | 711 (118)     | 542 (472–611)     |

*Similar caption letters mean no statistical difference.

**Figure 4** Staircase graphs for both groups. The red circle represents the beginning of the test. Survival (filled circle) and failure (empty circle) profiles observed during the fatigue test.

**Figure 5** Representative micrographs (SEM, 80× and 150×) of the fractured specimens from each group indicate the beginning of the fracture from the tensile surface. The region highlighted by the black half circle and indicated by the white pointers demonstrates the fracture origin (tensile side). White arrows signal the direction of crack propagation.
DISCUSSION

This study investigated if the Fast sintering mode (FM) would promote different characteristics for a 3Y-TZP zirconia compared to a Standard firing protocol (SM). The results rejected the null hypotheses since no difference was observed on the evaluated zirconia morphology, microstructure, mechanical strength and translucency parameter.

The use of prefabricated and calibrated programs without being able to modify the sintering parameters likely provided greater standardization in the topography, microstructure and physical properties used among the zirconia discs, regardless of the sintering mode. A previous study in which sintering programs were customized so that the temperature was increased and the time prolonged showed an increase in grain growth.\(^{11}\) Other study showed that the sintering time and temperature influence the average grain size of zirconia.\(^{14}\)

Although a prefabricated rapid sintering program was used in this study, it was possible to observe a similar result through high magnification micrographs. Even though the grain size was not measured in the present study, the sintering mode did not appear to have influenced the homogeneous grain size and distribution, as observed in the micrography images. Therefore, it is suggested for further studies to evaluate the influence of different fast sintering modes on the grain size and distribution. No difference on grain size of the 3Y-TZP grades using conventional or speed sintering mode has also been reported; which behaved differently for high translucent zirconia.\(^{13}\)

Despite the addition of oxides to stabilize the zirconia material and allow tetragonal conformation to be maintained at room temperature, this ceramic is still susceptible to phase transformation, color change and translucency changes. Its vulnerability is closely associated with the sintering conditions.\(^{11}\) Moreover, it was observed that all specimens mainly contained tetragonal phase with no phase transformation for either sintering mode regarding their crystalline composition. The XRD graphs corroborates with a previous study that investigated the same zirconia under different surface conditions, however they did use fast sintering protocols.\(^{21}\) Another investigation has found 15 wt.% of cubic phase for the same zirconia; however the author crystalized the zirconia using 1500°C for 2 h.\(^{22}\) Other study showed low amounts of cubic phase for other 3Y-TZP materials: 8 wt.% for CEREC Zirconia sintered using a Speed sintering (15 min/2 min/1578°C) and 20 wt.% for inCoris TZI sintered using a conventional protocol (4 h/2 h/1510°C).\(^{13}\) In addition, the authors found that the zirconia phase composition was influenced by sintering mode, since speed sintered zirconia presented more tetragonal phase (92.1%) than the conventionally sintered (80%). Therefore, further studies should be conducted to determine the influence of different fast sintering modes on the relative amount of transformed monoclinic, tetragonal and cubic phases (in %) on the surfaces, to corroborate or not with these findings.

The phase transformation is able to increase the surface roughness of stabilized tetragonal zirconia. However, the current investigation observed that the evaluated sintering modes did not affect the surface roughness or topography. High surface roughness has been related to biofilm formation since it accumulates more nutrients for and facilitates the microorganisms adherence. In addition, higher amount of adhered microorganisms is associated with an increased risk of caries,\(^{19}\) which can compromise the restoration success. In this investigation three different surface parameters have been used to identify differences in the surface profiles (Ra, Rz and RSm). The use of more than one parameter has been advocated since different surfaces can present similar roughness across a parameter and yet have distinct profiles when evaluated by an association of parameters. Thus, the first hypothesis that the fast sintering mode could influence the surface morphology and microstructure of a translucent monolithic zirconia ceramic was rejected.

Translucency parameter (TP and TP\(_{00}\)) is represented by the light behavior on a surface which allows its partial passage and dispersion. This study measured TP and TP\(_{00}\) based on the CIEL\(^*\)\(\alpha\)\(b\)\(^*\) and CIEDE 2000 color difference of samples with uniform thickness between a black and a white background.\(^{26,27,29}\) The results showed that the sintering process cannot lead to a change in translucency in the evaluated zirconia. Although the FM has a more homogeneous microstructure according to the micrographs presented in high resolution, it did not affect the TP or TP\(_{00}\) of the material, rejecting the third hypothesis. Similar finding was observed for 1.2 mm thickness yttria-partially stabilized zirconia (Y-PSZ), where no effect on translucency was observed between speed sintering (~90 min) and conventional sintering protocols (~7 h).\(^{10}\) However, the results differ from another study\(^{11}\) that identified an increase in the translucency of another monolithic translucent zirconia when sintered in only 10 minutes. The result was interpreted by the ceramic grain density and size being affected by the sintering method used. Other investigation used thinner specimens (0.5 mm) and observed that speed sintering decreased the TP compared to the conventional sintering mode for 3Y-TZP zirconia.\(^{13}\) According to the authors, the translucency of a 3Y-TZP does not allow to mimic natural teeth when its thickness is higher than 1 mm. And, that the low translucency is justified due to birefringence (at the grain boundaries or residual pores) or secondary phases.

The fact that the surface roughness is similar among the groups evaluated in this study also suggests that the surface characteristic is similar between the groups regardless of the sintering mode, which corroborates the findings of the fatigue test. The maintenance of the groups in the tetragonal phase also showed the same behavior under fatigue. Therefore, the second hypothesis that the fast sintering mode would affect the translucent zirconia mechanical strength was rejected. Fatigue failure can be defined as material fracture due to slow crack propagation under low intensity repeated cyclic stresses.\(^{24}\) Thus, mechanical tests such as maximum fracture load and mechanical fatigue tests have been developed to better understand these failures, with an evaluation of the material or structure resistance to a specific strength type and intensity being the purpose of these tests. Speed sintering applies rapid temperature changes and the thermal shock could lead to surface-crack formation and subsequent material fracture, factor that can affect the materials fatigue behavior.\(^{13}\) Earlier in vitro studies have shown that the sintering temperature and
holding time can affect the flexural strength of translucent monolithic zirconia. Although it has been reported that a prolonged sintering time increases the flexural strength of a translucent monolithic zirconia as a function of grain size variation and phase transformation,\textsuperscript{15} the results showed similar results among the fast and standard sintering modes. This corroborates other findings,\textsuperscript{10,14} in which the different sintering parameters had no effect on the biaxial flexural strength. Complementing the in vitro mechanical tests, fractography is an important method to identify the fracture origin and its characteristics to determine the possible failure causes.\textsuperscript{26} The fracture patterns and failure origin were similar by the fractographic analysis, always starting from the tensile side, as observed in previous studies.\textsuperscript{9,15,24}

The current study compared two sintering modes of a zirconia sintered in a new sintering furnace, however other studies should be conducted to test the effects when using other programs or equipment. Also, further investigations need to be performed focusing on the influence of time and sintering mode on distinct zirconia ceramics. As limitations of this study, the grain size and relative amount of transformed monoclinic, tetragonal and cubic phases (%) or the performance of the evaluated fast sintered zirconia under different condition, for example, adhesively cemented or veneered, were not performed. In addition, the lack of standardization for specimens thickness,\textsuperscript{30} firing protocols and data analysis makes difficult the comparison of translucency parameter with other studies.

5 | CONCLUSION

Within the limitations of this current study, it was concluded that:

- The fast sintering mode did not affect the surface morphology or microstructure of a translucent zirconia;
- The evaluated zirconia fatigue performance was similar when sintered using standard and fast sintering modes;
- The fast sintering mode did not affect translucency of the evaluated monolithic zirconia.

DISCLOSURE

The authors declare that they do not have any financial interest in the companies whose materials are included in this article.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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