https://doi.org/10.4047/jap.2021.13.6.385

Mechanical behavior and microstructural characterization of different zirconia polycrystals in different thicknesses

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PURPOSE. To characterize the microstructure of three yttria partially stabilized zirconia ceramics and to compare their hardness, indentation fracture resistance (IFR), biaxial flexural strength (BFS), and fatigue flexural strength. **MATERIALS AND METHODS.** Disc-shaped specimens were obtained from 3Y-TZP (Vita YZ HT), 4Y-PSZ (Vita YZ ST) and 5Y-PSZ (Vita YZ XT), following the ISO 6872/2015 guidelines for BFS testing (final dimensions of 12 mm in diameter, 0.7 and 1.2 ± 0.1 mm in thicknesses). Energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses were performed, and mechanical properties were assessed by Vickers hardness, IFR, quasi-static BFS and fatigue tests. **RESULTS.** All ceramics showed similar chemical compositions, but mainly differed in the amount of yttria, which was higher as the amount of cubic phase in the diffractogram (5Y-PSZ > 4Y-PSZ > 3Y-TZP). The 4Y- and 5Y-PSZ specimens showed surface defects under SEM, while 3Y-TZP exhibited greater grain uniformity on the surface. 5Y-PSZ and 3Y-TZP presented the highest hardness values, while 3Y-TZP was higher than 4Y- and 5Y-PSZ with regard to the IFR. The 5Y-PSZ specimen (0.7 and 1.2 mm) showed the worst mechanical performance (fatigue BFS and cycles until failure), while 3Y-TZP and 4Y-PSZ presented statistically similar values, higher than 5Y-PSZ for both thicknesses (0.7 and 1.2 mm). Moreover, 3Y-TZP showed the highest (1.2 mm group) and the lowest (0.7 mm group) degradation percentage, and 5Y-PSZ had higher strength degradation than 4Y-PSZ group. **CONCLUSION.** Despite the microstructural differences, 4Y-PSZ and 3Y-TZP had similar fatigue behavior regardless of thickness. 5Y-PSZ had the lowest mechanical performance. [J Adv Prosthodont 2021;13:385-95]

KEYWORDS

Dental ceramics; Mechanical stress; Y-TZP ceramic; Step-stress accelerated fatigue test; Material thickness

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Received September 10, 2021 / **Last Revision** December 2, 2021 / **Accepted** December 10, 2021

INTRODUCTION

Mechanical properties such as mechanical strength, hardness, fracture toughness and aesthetics, bond strength to resin, and biological properties of yttria partially stabilized zirconia (Y-PSZ) are already well-established in the scientific literature due to excellent results found in laboratory studies, literature reviews and clinical trials.[1-6] More translucent zirconia has recently emerged to address the optical deficiency of conventional zirconia.[7]

The opaque appearance of zirconia is the result of the interaction of grain size (approximately 0.4 µm) with light wavelength (between 0.1 and 0.7 µm), and the incompatibility of the grain refraction index of the different phases (monoclinic, cubic and tetragonal). These factors spread light rather than transmit it through the material.[7-9] On the other hand, translucent zirconia has smaller grain sizes and the refraction indexes of these grains and crystalline matrix are closer,[2] which can lead to more similar translucency levels to those of glass ceramics.[5] In addition, a greater amount of yttria (stabilizing oxide) is included, with a consequent higher cubic phase formation. However, with the increase in translucency due to changes in microstructure and composition, the mechanical properties of materials can be affected.[7] As an example, the phase transformation may not occur if the grains are smaller than 0.2 µm, which leads to a decrease in fracture toughness.[1]

Regarding translucent zirconia fatigue resistance, high translucency (or second generation) zirconia has higher fatigue resistance compared to feldspathic, polymer-infiltrated, silicate and lithium disilicate ceramics.[10] Even more translucent zirconia has recently been produced, namely 4Y-PSZ and 5Y-PSZ, known as extra and ultra-translucent zirconia, respectively, or third generation zirconia. However, little is known about their fatigue resistance so far, which is of uttermost importance in clinics, mainly because these materials tend to be indicated for multiple-unit restorations which must support a great amount of stress.

On the other hand, degradation at low temperature (LTD) can influence the behavior of zirconia from different generations, as was verified after aging in an autoclave to affect the mechanical properties of the first and second-generation types.[11] New fully stabilized zirconia ceramics seem totally inert to aging in an autoclave, being able to avoid LTD; however, it is still unclear if the stimuli found in the clinical environment, mainly in the presence of moisture and loading, could promote degradation of these materials.[12]

Clinical fracture of ceramic materials usually occurs under loads far below their fracture strength due to fatigue corrosion/degradation via slow crack growth or cyclic fatigue mechanisms.[13,14] The ceramic thickness clinically influences both translucency and strength. Thus, the thinner the ceramic restoration, the more prone it will be to flexural damage.[15]

Thus, the objective of this work was to compare 3Y-TZP (Yttria-tetragonal stabilized zirconia), 4Y-PSZ and 5Y-PSZ (Yttria-parcially stabilized zirconia) in terms of microstructure and mechanical properties (hardness, indentation fracture resistance, flexural and fatigue strength). The null hypothesis of this study is that 3Y-TZP, 4Y-PSZ and 5Y-PSZ with distinct thicknesses will have similar microstructure and mechanical properties.

MATERIALS AND METHODS

Zirconia ceramic blocs of 3Y-TZP (Vita YZ HT, Vita Zahnfabrik, Bad Säckingen, Germany), 4Y-PSZ (Vita YZ ST, Vita Zahnfabrik) and 5Y-PSZ (Vita YZ XT, Vita Zahnfabrik) were machined in pre-sintered form to obtain cylinders of 15 mm in diameter. Circular sections of the three ceramics groups (N = 318) were obtained by a cutting machine (Isomet 1000; Buehler, Lake Bluff, IL, USA) under water cooling, with initial dimensions around of 1.65 mm and 1 mm thickness and 15 mm in diameter. The discs were manually polished with #1200 SiC sandpaper under water to remove irregularities inherent to the cut and the thickness was controlled with a digital caliper.

All specimens (3Y-TZP, 4Y-PSZ and 5Y-PSZ) were sintered in a speed oven (inFire HTC; Dentsply Sirona, Charlotte, NC, USA) according to the manufacturer's instructions: initial temperature of 25°C, heating rates of 17, 8 and 4°C/min and remaining for 120 min to 1450, 1530 and 1450°C, respectively. Each ceramic group (n = 106) had final subgroups with thickness of 0.7 ± 0.1 mm (minimum thickness recommended by the manufacturer of 5Y-PSZ) (n = 53) and of 1.2 ± 0.1 mm (n = 53), both with 12 mm in diameter (according to ISO 6872/2015) to evaluate the thickness effect on the biaxial flexural strength (quasi-static and fatigue tests).

The specimens of each ceramic (n = 3), regardless of thickness, were analyzed for surface morphology and characteristics of zirconia grains using a scanning electron microscopic with high-resolution field emission (FEG-SEM) (Mira 3; Tescan, Brno, Czech Republic) in 20,000× and 100,000× magnification. A chemical analysis by energy-dispersive X-ray spectroscopy (EDX) (Bruker Nano GmbH, Berlin, Germany) coupled to the SEM was also carried out to identify the surface chemical microconstituents.

Additional specimens of each ceramic (n = 3), regardless of thickness, were analyzed by X-ray diffraction (XRD; Philips X'pert PRO MRD, Almelo, Netherlands) to identify the zirconia crystallization pattern. An X-ray diffractometer (EMPYREAN - PANalytical) was used, equipped with CuKα (λ = 1.5418), operating at 40 kV and 40 mA in the range of $10° \leq 2\theta \leq 90°$, $\Delta\theta = 0.02°$ and time for $\Delta\theta$ of 30 seconds. The XRD data were evaluated by identifying the crystalline phases after comparing the experimental spectra with standard diffraction spectra from the JCPDS (Joint Committee on Powder Diffraction Standards) and ICSD (Inorganic Crystal Structure) databases. The HighScore software program (Philips X'pert MPD; PANalytical, Almelo, Netherlands) helped with the assignments of the spectra.

A micro hardness tester was used to assess the Vickers hardness and fracture toughness of each ceramic group regardless of thickness. Each group (n = 10) was indented with a Vickers diamond tip in the center of each disc with a load of 9.8 N for 10 seconds on the micro hardness tester (HMV-G21DT; Shimadzu, Singapore), and the marking diagonals were measured. Vickers hardness (HV) was calculated using the following equation:

$$HV = \frac{1.8544P}{d^2}$$

in which P is the load and d is the mean of the indentation diagonal measurements.

To determine fracture toughness, the specimens (n = 15) were also indented with a Vickers diamond with a load of 19.61 N for 12 seconds. The marking diagonals and the extension of the surface radial cracks were measured using the following equation proposed by Anstis[16]:

$$K_{Ic} = k\left(\frac{EH}{H}\right)^{0.5} \times \frac{P}{C^{3/2}}$$

in which E is the modulus of elasticity, P is the applied load (in N), H is the Vickers hardness, c is the average length of the radial crack measured from the center of the indentation (in m) and k is a constant equal to 0.016. The values used for E in the manufacturer's specifications are 210 GPa for the three types of zirconia.

The specimens of each thickness group (n = 10) were tested according to ISO 6872/2015 guidelines using a universal testing machine (Emic DL-1000; Emic, São José dos Pinhais, PR, Brazil) to determine the quasi-static biaxial flexural strength. Thus, it was possible to determine the loading profile to be used during the fatigue test.

Then, the additional specimens (n = 15) were submitted to a Fatigue Biaxial Flexural Strength test in an electrodynamic machine (Instron ElectroPlus E3000; Instron Corporation, Norwood, MA, USA) according to ISO 6872 (2015) for the biaxial flexural strength test (piston-on-three-balls). The assembly was immersed in distilled water, and a flat circular tungsten piston (Ø = 1.6 mm) was used to apply the load.

The step-stress fatigue method was performed with a frequency of 20 Hz, always considering a minimum tension of 10 MPa and the maximum tension desired for each stage of each cycle. Each specimen was submitted to incremental steps of stress under a predetermined number of cycles, initially 5000 cycles under 200 MPa (maximum applied stress on this step) to accommodate piston/specimen relation, and then additional incremental steps of 25 MPa for 10,000 cycles starting from 400 MPa until the complete failure (fracture) of the specimen. The step (MPa) at the failure and the number of cycles required for failure were recorded for each tested specimen. It is noteworthy that the equations presented in ISO 6872 (2015) were used to determine the amount of load necessary to apply the desired stress for each specimen in each step (i.e. maximum applied stress on each step).

Representative fractured specimens (n = 5) were evaluated in a stereomicroscope (Discovery V20; Zeiss, Jena, Germany) at 25× magnification to observe the fracture characteristics, and then the representative specimens (n = 3) were chosen from each group to analyze using a scanning electron microscope (FEG-SEM) (Inspect S50; FEI Company, Brno, Czech republic). The specimens were gold sputtered, and the acceleration voltage used was 15 kV for fractography and 20 kV for surface images. The images were observed in secondary electrons and backscattered electrons at low and high magnification.

The Kolmogorov-Smirnov, Shapiro-Wilk and Levene Test (95%) normality tests were performed for hardness, indentation fracture resistance and flexural strength data, which showed that the data were normal and homoscedastic ($P > .05$). Two-way ANOVA and Tukey tests were applied for quasi-static biaxial flexural strength data, while Kaplan-Meier and Mantel-Cox tests (log-rank) were run for fatigue data using the SPSS Statistics software program (IBM, Armonk, NY, USA). The Vickers hardness and indentation fracture resistance data obtained were submitted to one-way ANOVA and the Tukey Post-hoc test (Minitab 19 software; Minitab Inc., State College, PA, USA). A 5% significant level was used for all analyses.

The strength degradation (%) was calculated through the decrease percentage of the quasi-static and fatigue strength tested specimens.[17]

RESULTS

The FEG-SEM images (Fig. 1) show that the micro-

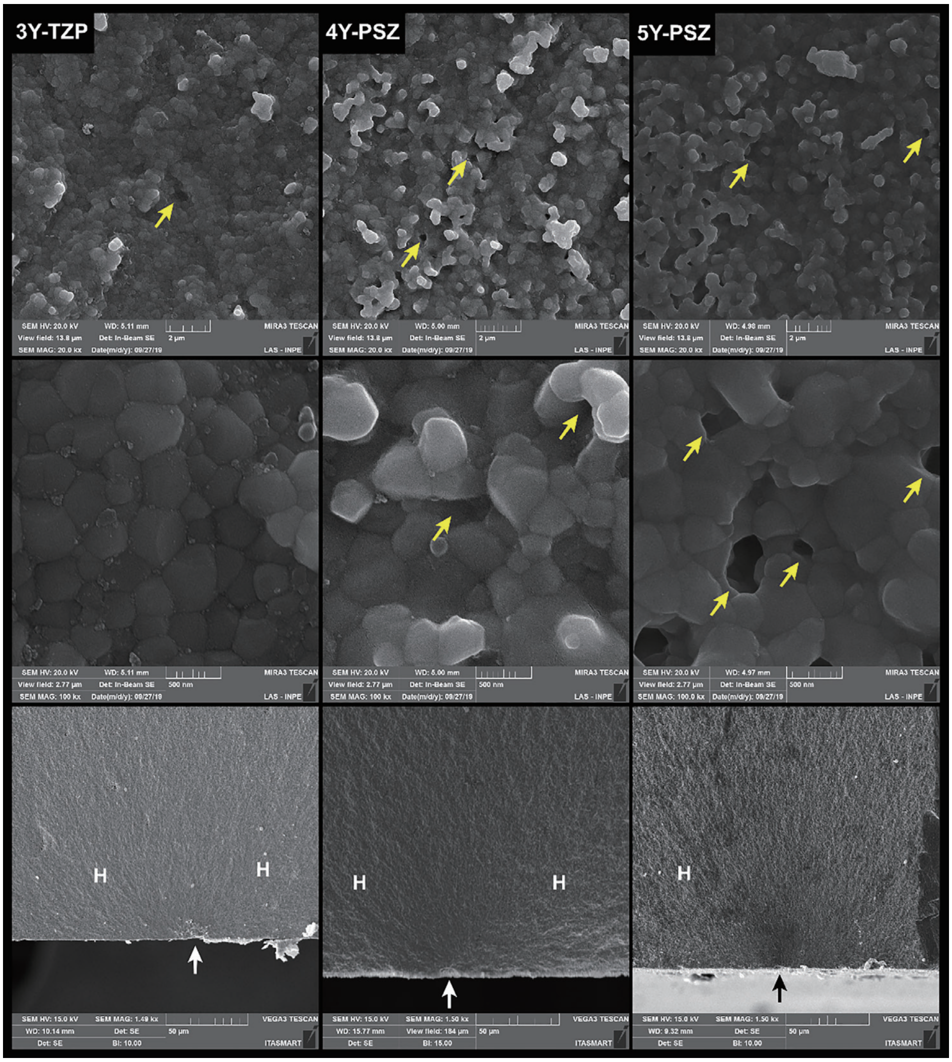

Fig. 1. Topography micrographs of 3Y-TZP (left), 4Y-PSZ (center) and 5Y-PSZ (right column) ceramics under 2000 × magnification in the first row, 10000× in the second row, and the fracture surfaces in the third row. It possible to observe the grain morphology and the presence of pores, mainly in 4Y- and 5Y-PSZ ceramics (yellow arrows indicate pores/surface defects). Regarding the fracture patterns, all images were observed in secondary electrons, the specimen was submitted to tensile stress at the bottom and compression stress at the top – arrows indicate the fracture origin, and H indicates hackles.

structures are slightly different. It is noted that the 4Y-PSZ and 5Y-PSZ zirconia present more pores on the surface, acting as defects in the material, while 3Y-TZP zirconia exhibits fewer surface defects, greater densification, and a more uniform aspect of the grains.

The EDX analyses for the chemical composition demonstrated that the more translucent the zirconia (branding indication), the greater the yttrium content (5Y > 4Y > 3Y). The amount of zirconium oxide element decreases according to the increase in material translucency (Table 1).

The XRD spectra and quantification of the crystalline phases analyses (Fig. 2) show that more translucent zirconia has higher amounts of cubic phase and consequently less amount of tetragonal phase (c-phase: 5Y-PSZ > 4Y-PSZ > 3Y-TZP).

Table 1. Quantification of the chemical elements by mass (%) present in the zirconia ceramics according to EDX analysis

Material	Zr	Y
3Y-TZP	62.73 %	8.03 %
4Y-PSZ	61.95 %	10.13 %
5Y-PSZ	58.30 %	12.36 %

Zr= Zirconium and Y= Yttrium

The materials present statistically significant differences in hardness ($P = .001$) and in indentation fracture resistance ($P = .005$) by the one-way ANOVA test (95%) (Table 2). The 5Y-PSZ zirconia has the highest hardness value, while 4Y-PSZ showed the lowest value. The 3Y-TZP zirconia is tougher than 4Y-PSZ and 5Y-PSZ, which are similar.

The quasi-static biaxial flexural strength data are shown in Table 2. The two-way ANOVA showed that the ceramics factor has a statistically significant effect ($P = .036$), while the thickness factor ($P = .157$) and the interaction ($P = .119$) have no effect. As shown in Table 2, Tukey's test shows that the 4Y-PSZ showed the highest resistance value and the 5Y-PSZ showed the lower resistance values to biaxial flexural strength.

The 5Y-PSZ zirconia statistically presented the lowest fatigue strength and number of cycles until failure (Table 3). The 3Y-TZP and 4Y-PSZ showed similar fatigue behavior. The strength degradation (Table 2)

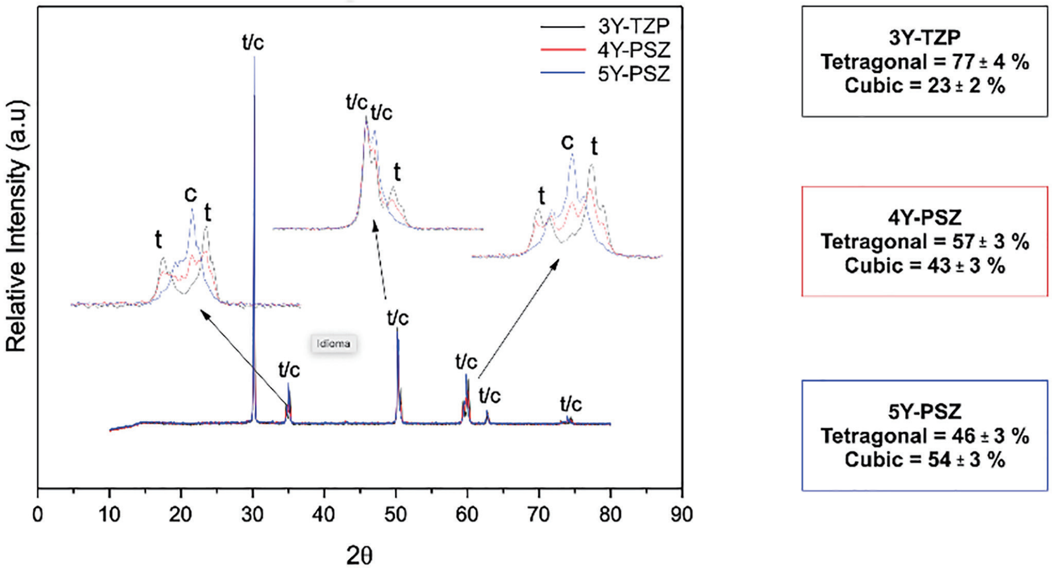

Fig. 2. XRD graphs depicting the peaks related to each specific crystallographic phase, enabling to infer the material's crystallographic microstructure. The letter "t" indicates the peaks of the tetragonal phase and letter "c" the peaks of the cubic phase. The squares show the quantification of the tetragonal and cubic phases according to Rietveld's analysis.

Table 2. Mean values and standard deviation of quasi-static biaxial flexural strength, strength degradation percentage, Vickers hardness, and indentation fracture resistance

Material	Thickness (mm)	Flexural strength (MPa)	Strength degradation (%)	Vickers Hardness (HV)	Indentation Fracture Resistance (MPa.mm$^{1/2}$)
3Y-TZP	0.7	741.3 ± 78.2^{AB}	29.85	1421.5 ± 109.7^{AB}	5.66 ± 0.56^{A}
	1.2	975.7 ± 132.7^{AB}	47.04		
4Y-PSZ	0.7	930.9 ± 326^{A}	35.19	1314.6 ± 80.1^{A}	4.44 ± 0.58^{B}
	1.2	889.1 ± 202.7^{A}	38.52		
5Y-PSZ	0.7	702.6 ± 133.6^{B}	46.38	1529.8 ± 152.6^{B}	4.29 ± 0.38^{B}
	1.2	750.7 ± 127.2^{B}	41.38		

*Two-way ANOVA and Tukey's post-hoc test (95%) for flexural strength data analysis, and One-way ANOVA and Tukey's test for hardness and indentation fracture resistance analysis (95%).

**Different letters represent the statistical differences in the columns.

Table 3. Results of fatigue biaxial flexural strength in MPa, the number of cycles until failure and respective confidence intervals (CI)

Groups	Fatigue biaxial flexural strength (MPa)	95% CI	Cycles until failure	95% CI
3Y-TZP 0.7	520.0 ^{AB}	467.7 - 572.2	89 × $10^{3\ AB}$	(78.5 - 99.4) × 10^3
3Y-TZP 1.2	516.7 ^{B}	442.1 - 591.1	88.3 × $10^{3\ B}$	(73.4 - 103.2) × 10^3
4Y-PSZ 0.7	603.3 ^{A}	568.2 - 638.4	105.6 × $10^{3\ A}$	(98.6 - 112.6) × 10^3
4Y-PSZ 1.2	546.6 ^{AB}	504.4 - 588.8	94.3 × $10^{3\ AB}$	(85.8 - 102.7) × 10^3
5Y-PSZ 0.7	376.7 ^{D}	334.0 - 419.2	60.3 × $10^{3\ D}$	(51.8 - 68.8) × 10^3
5Y-PSZ 1.2	440.0 ^{C}	407.9 - 472.0	73 × $10^{3\ C}$	(66.5 - 79.4) × 10^3

*Different letters indicate statistical differences in each column according to Kaplan-Meier and Mantel-Cox tests ($\alpha = 0.05$).

shows that 3Y-TZP of 1.2 mm and 0.7 mm suffered the highest and lowest percentage of degradation, respectively. Regarding these results, it was also possible to observe that 5Y-PSZ had higher strength degradation than 4Y-PSZ group.

The survival rates (Table 4) corroborate the fatigue findings, i.e., the 3Y-TZP and 4Y-PSZ lasted a longer time until failure (higher survival rates), while 5Y-PSZ had earlier fracture (lower survival rates).

The FEG-SEM images (Fig. 1) show the failures started on the side subjected to tensile stresses, always with a defect which originated from the fracture on the disc surface. It was possible to observe that the 5Y-PSZ samples always fracture in more pieces than 3Y-TZP and 4Y-PSZ.

DISCUSSION

The results showed significant differences for both microstructural characteristics and for the mechanical properties; therefore, the null hypothesis was rejected. The 5Y-PSZ contains a higher ratio of cubic phase and had the worst fatigue behavior compared to the other two zirconia ceramics. The zirconia with lower percentage of cubic phase and higher of tetragonal phase (3Y-TZP) was tougher than the other two.

Microstructure is one of the factors, which can influence the physical and optical properties of a ceramic.[2,18] In addition to knowing the surface morphology and the size and characteristics of the grains, it is possible to identify problems in processing zirconia ceramics through observing the microstructure. The three translucent zirconia ceramics in the present study showed slight differences among the grains; 4Y- and 5Y-PSZ showed the presence of superficial pores, while 3Y-TZP showed fewer surface defects (Fig. 1). The defects can be inherent (included during the material processing) or a result from a pullout of surface

Table 4. Survival rates considering data of fatigue strength and number of cycles until failure (probability of exceeding strength and number of cycles without fail and their respective standard deviations)

Groups	Strength (MPa) / Cycles																		
	100/ 5.000	150/ 15.000	200/ 25.000	250/ 35.000	300/ 45.000	350/ 55.000	400/ 65.000	450/ 75.000	500/ 85.000	550/ 95.000	600/ 105.000	650/ 115.000	700/ 125.000	750/ 135.000	800/ 145.000	850/ 155.000	900/ 165.000	950/ 175.000	1000/ 185.000
3Y-TZP 0.7	1	1	1	1	1	0.86 (0.09)	0.86 (0.09)	0.73 (0. 11)	0.47 (0.13)	0.20 (0.10)	0.13 (0.09)	0.07 (0.06)	0.07 (0.06)	0.00	-	-	-	-	-
3Y-TZP 1.2	1	1	1	1	1	0.80 (0.10)	0.73 (0.11)	0.73 (0.11)	0.40 (0.13)	0.20 (0.10)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.07 (0.06)	0.00	-
4Y-PSZ 0.7	1	1	1	1	1	1	1	1	0.80 (0.10)	0.67 (0.12)	0.47 (0.13)	0.13 (0.09)	0.00	-	-	-	-	-	-
4Y-PSZ 1.2	1	1	1	1	1	1	0.93 (0.06)	0.80 (0.10)	0.60 (0.13)	0.33 (0.12)	0.20 (0.10)	0.07 (0.06)	0.00	-	-	-	-	-	-
5Y-PSZ 0.7	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.73 (0.11)	0.67 (0.12)	0.33 (0.12)	0.00	-	-	-	-	-	-	-	-	-	-	-
5Y-PSZ 1.2	1	1	1	1	0.93 (0.06)	0.80 (0.10)	0.73 (0.11)	0.33 (0.12)	0.00	-	-	-	-	-	-	-	-	-	-

grains during sample preparation. Regardless of the origin of the defects, this may reflect a deficient industrial process for these ceramics.[19]

The yttria amounts in zirconia have been increased to improve optical properties, which are also attributed to the increase in the cubic phase.[2,6,7,20,21] The increase in cubic phase from 3Y-TZP to 4Y-PSZ is 20% and 31% to 5Y-PSZ, accompanied by a 2.1% increase of yttria from 3Y-TZP to 4Y-PSZ and 4.3% from 3Y-TZP to 5Y-PSZ (Fig. 2), according to other studies.[22,23] This increase is due to the larger amount of stabilizing oxides in zirconia (yttria), which leads to a greater amount of cubic phase and larger grains.[24] It can be noted that both the increased grain size and the reduced stabilizer content (yttria) can cause greater susceptibility to low temperature degradation (LTD).[25,26] Therefore, an improved stabilization mechanism with yttria can prevent LTD, but this needs further investigation.

Harianawala *et al*.[3] showed that a translucent zirconia had more transmittance than a conventional one, which can be attributed to the manufacturing procedures of these ceramics since both are 3Y-TZP. A denser, less porous and more translucent zirconia may be obtained depending on the industrial processing, either hot isostatic pressing, microwave sintering or spark plasma sintering.[27-29] Polycrystalline ceramics show high transmittance values when zirconia grains are smaller and uniform with minimal porosity.[29] The existence of residual pores after sintering at high temperatures causes significant incident light dispersion, deteriorating the optical properties of the material.[30]

One study in which the three translucent zirconia ceramics (3Y-TZP, 4Y-PSZ and 5Y-PSZ) were evaluated showed that the microstructures were similar and no porosity was observed in the materials. Moreover, the grain boundaries were clearly visible, but the grains were not uniform, showing smaller grains for 3Y-TZP (437 ± 40 nm) and larger grains for 5Y-PSZ (815 ± 97 nm).[23] This difference was not observed herein, as the images showed similar grain sizes (Fig. 1).

The microstructure can assist in estimating the lifetime and clinical performance of the restorations, constituting factors which can determine the indication and material selection.[1,31,32] However, in addition to the microstructural characteristics, it is also important to know and evaluate the mechanical properties to predict how the materials will behave under complex situations, especially under cyclical loads and the presence of moisture.[13]

The accelerated fatigue tests were developed to analyze the lifetime of the materials so that the samples are submitted to stresses greater than those found during chewing, but lower than the quasi-static fracture test, such as biaxial flexure tests.[33,34] Step-stress testing stands out among the fatigue methods due to optimizing the time needed to perform the test, for incorporating survival analysis and for estimating the lifetime and the accumulation of damage in the materials.[13,35] The stress applied in this test to the specimen is increased until failure or suspension occurs (threshold survival). There were no chipping failures in this study, so only catastrophic fractures were considered as failures. A light load profile with steps of 25 MPa step size was used, considering a sensitive way of identifying possible small changes through different material conditions.[35,36] Another aspect in relation to the fatigue test is the frequency. A 20 Hz frequency was used in this study, according to Fraga *et al*.,[37] and this high frequency did not alter the fatigue behavior of zirconia-based ceramics.

The 5Y-PSZ zirconia in thin thickness of 0.7 mm showed the worst fatigue strength, while 3Y-TZP and 4Y-PSZ were similar and better (Table 3). This can be caused by the amount of tetragonal and cubic phase, as the more translucent zirconia ceramic such as 5Y-PSZ present a greater amount of cubic phase, but a smaller amount of tetragonal phase, thus affecting the transformation toughening mechanism, which negatively affects the mechanical properties.[12]

The sintering parameters can influence the microstructural, optical and mechanical properties.[38-40] The 4Y-PSZ zirconia is sintered at a final temperature of 1530°C, while the maximum temperature for 5Y-PSZ is 1450°C, which enables listing the best results of 4Y-PSZ due to the higher temperature. Grambow *et al*.[38] compared several mechanical properties of these same zirconia ceramics in the range of 1400 - 1600°C sintering temperatures, showing that while the temperature decreased the mechanical properties such as biaxial flexural strength (quasi-static and dynamic loading test), the Vickers hardness significantly increased, being greater for 4Y-PSZ than for 5Y-PSZ.

The thickness of a restoration has been shown to influence both the mechanical strength and stress distribution. A recent study by Dal Piva *et al*.[41] showed that thinner crowns presented higher stress peaks than thicker ones, and the stress concentration regions for all situations were compatible with the tensile strength generated in response to the load application, as well as in the intaglio surface. In addition, the ceramic thickness also affected the translucency, taking into account that the fracture load will be reduced when reducing the dimensions of a restoration.[42] In this study, the thickness of the samples was not a relevant factor for the quasi-static biaxial flexural strength load (Table 2) because the equations used to calculate the fracture stress already took into account the sample volume; however, it was a relevant factor for fatigue life of 5Y-PSZ, as the thin thickness showed lower fatigue resistance and survival rates. Moreover, Alraheam *et al*.[43] showed that regardless of the type of zirconia, the reduction in thickness decreases the fracture load of biaxial flexural strength.

Regarding the strength degradation, 5Y-PSZ showed a higher mechanical degradation percentage compared to 4Y-PSZ and 3Y-TZP (0.7 mm). As previously mentioned, the most translucent zirconia (regarding the manufacturer names) have more surface defects (5Y-PSZ > 4Y-PSZ > 3Y-TZP), which can cause greater susceptibility to stress corrosion by negatively affecting the Zr-O bonds in the presence of moisture, contributing to mechanical degradation. The only exception was 1.2 mm 3Y-TZP zirconia, which showed the highest strength degradation percentage; this may be related to either the materials thickness, as larger volumes lead to a greater probability of defects and consequently greater susceptibility to stress corrosion; or the susceptibility to cyclical fatigue that seems to be associated with transformation toughening, which is greater in 3Y-TZP due to the greater amount of tetragonal phase.[17] The degradation mechanisms involve several aspects and must be further studied so that these mechanisms can be defined for each type of ceramic and its microstructure.

The hardness of a material is related to stiffness and mechanical strength. During the beginning of crack (which may be caused by surface flaws), it is possible that this resistance of the material to plastic deformation improves the clinical behavior of the restoration.[36] In this study, the 5Y-PSZ and 3Y-TZP zirconia ceramics showed the highest hardness value, while the 4Y-PSZ showed the lowest hardness (Table 2). Sen and Isler[23] evaluated the hardness values of the translucent zirconia ceramics and found no significant difference. The authors even reported the difficulty of comparing data and the inconsistency of the technique, which is dependent on surface polishing, grain sizes, quantity of each of the phases, etc. In contrast, other studies have evaluated the phase content, the final sintering temperatures and the different grain sizes of the zirconia ceramics, also showing significant differences in the hardness values according to these properties.[44,45]

There were also statistically significant differences in indentation fracture resistance in the present study, mainly that 3Y-TZP showed greater fracture toughness (Table 2). On the other hand, the fracture toughness results in this study were greater than the results obtained in the work of Sen and Isler,[23] presenting values of 4.27 ± 0.79 MPa·mm$^{1/2}$ for 3Y-TZP, 3.78 ± 0.56 MPa·mm$^{1/2}$ for 4Y-PSZ and 3.14 ± 0.37 MPa·mm$^{1/2}$ for 5Y-PSZ; this can be caused by the minor defects observed in the SEM micrographs of the three translucent zirconia studied (Fig. 1), since the crack starts with the largest defect spreading to the smallest defects. Then, since the crack starts at the biggest defect and spreads to the smallest defects, it generates a toughening effect which prevents its spread, as it reaches an empty space which prevents the crack from continuing. We are aware of the limitations regarding the indentation method related to residual stresses, but this methodology still presents great acceptance among material scientists.[23,46]

Thus far, several zirconia materials for monolithic applications have been evaluated in terms of the mechanical properties and the translucency. Kwon *et al*.[5] showed that 5Y-PSZ (called ultra-translucent multilayer zirconia by the manufacturer) has significantly higher flexural strength when compared to another type of ceramic such as lithium disilicate, but has significantly less translucency. Another recent study evaluated translucent 5-PSZs against a conventional zirconia and concluded that the properties of each translucent material depend on the material. However, one 5-PSZ showed greater flexural strength and fracture resistance compared to the others.[47]

The crack origin and direction of its propagation can be clearly observed in a fractographic analysis, in addition to the presence of porosities and inclusions during the analysis.[48,49] In the present study, 5Y-PSZ presented the lowest fracture strength, which is probably related to the greater number of pores, as demonstrated in the fractography by a large round defect (Fig. 1). One study analyzed 3 types of Y-TZP zirconia, showing that the zirconia with lower biaxial flexural strength showed greater porosity.[50]

Even though this *in vitro* study has some inherent limitations, such as the use of non-anatomic specimens, the axial load application and the use of an accelerated life test, it was demonstrate that zirconia with high amount of yttria and thinner thicknesses (5Y-PSZ) could be indicated for prosthodontics restorations under low loads. Despite the microstructural differences, zirconia with lower yttria's content (4Y-PSZ and 3Y-TZP), regardless of thicknesses, is indicated for the restoration under strong masticatory load. Thus, further investigations are needed to analyze translucent zirconia in different conditions such as the use of anatomical specimens, behavior under different surface treatments and when the ceramic material is adhesively cemented to the dental substrate.

CONCLUSION

Within certain limitations, it can be concluded that the changes in the microstructure from 3Y-TZP to 4Y- and 5Y-PSZ, as well as the increase in the amount of yttria, led to decreases in mechanical properties (flexural strength, fatigue strength, hardness and indentation fracture resistance). Besides that, zirconia polycrystals with higher tetragonal-phase content and lower cubic-phase content presented better fatigue behavior.

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